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<p>(54) Title: ELECTROLUMINESCENT DEVICES</p> <p>(57) Abstract</p> <p>An electroluminescent device has at least two active semiconductive conjugated polymer light emitting layers arranged between a cathode and an anode. The layers are arranged so that at least part of at least two layers lie in an emission zone of the device.</p>		

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Title of the InventionELECTROLUMINESCENT DEVICESField of the Invention

This invention relates to electroluminescent devices and particularly to such devices which have a conjugated polymer as the light emissive layer.

Background to the Invention

Electroluminescent devices of the type with which the present invention is concerned are described for example in PCT/WO90/13148. Reference may also be made to articles by Burroughes et al in Nature (1990) 347,539 and by Braun and Heeger Applied Physics Letters (1991) 58,1982.

These devices offer potential as large-area flat-panel displays since they can be fabricated over large areas using solution-processing techniques. The basic structure of these electroluminescent (EL) devices comprises a polymer film sandwiched between two electrodes, one of which injects electrons, the other of which injects holes.

In the Nature reference the importance of balancing electron and hole injection rates through selection of charge injection electrodes is recognised. For these polymers, it is clear that injection and transport of electrons is less easy to achieve than for holes; this was indicated by the demonstration of improved device efficiencies when low work function metals such as calcium were used as the negative contact layer, as explained in the article in Applied Physics Letters. From photoluminescence studies it has been identified that an important non-radiative decay channel for excitons in these polymers is by exciton diffusion to charged defects which act as quenching sites. Metal injection electrodes can provide many defect states and efficiencies can

be raised substantially by introducing an additional layer between the emissive (polymer) layer and the calcium (electrode) layer. For this, a molecular semiconductor, 2-(4-biphenyl)-5-(4-tert-butylphenyl)-1,3,4-oxadiazole (butyl PBD) in a poly(methyl methacrylate) PMMA matrix has been used. This layer served both to prevent exciton migration to the metal contact and to enhance electron injection. In this context, reference is made to "Light-Emitting Diodes Based on Conjugated Polymers: Control of Colour and Efficiency", P.L. Burn, A.B. Holmes, A. Kraft, A.R. Brown, D.D.C. Bradley and R.H. Friend, Symposium N, MRS Fall Meeting, Boston Dec. 1991, MRS Symposium Proceedings 247, 647-654 (1992).

As described for example in PCT/WO92/03490, the contents of which are incorporated herein by reference, PPV can be chemically-modified to control its bandgap. For example, poly(2,5-dialkoxyphenylenevinylene) is red-shifted, by some 0.4eV, with respect to PPV. Copolymers of PPV and poly(2,5-dimethoxy-p-phenylenevinylene), PDMeOPV, allow fine-tuning of the band gap. Furthermore, controlled elimination of precursor leaving-groups allows both red- and blue- shifting of the gap with respect to that for PPV; the latter is achieved by interruption of conjugation along the chain by the presence of non-conjugated groups.

To date therefore it has been possible to have a limited amount of control over the colour of light emitted from an electroluminescent device using conjugated polymers. The present invention seeks to provide an electroluminescent device having a broader range of colour emission.

This has not been achieved in an EL device using conjugated polymer layers and is not a simple matter since the inventors have found that it requires that at least two conjugated polymer layers be put down and be simultaneously excited to emit radiation without one having a detrimental effect on the other.

Reference is made to EP-A-0443861 to Sumitomo which discloses electroluminescent devices made with two layers of conjugated polymers. In this device, only one layer is excited to emit radiation and the other layer is used as a charge transport layer to enhance the transfer of charges into the light emitting layer.

Summary of the Invention

According to one aspect of the present invention there is provided an electroluminescent device comprising: a first charge carrier injecting layer for injecting positive charge carriers; a first layer of a semiconductive conjugated polymer having a band gap selected such that when it is excited radiation at a first wavelength is emitted; a second layer of a semiconductive conjugated polymer having a band gap selected such that when it is excited radiation at a second wavelength is emitted; a second charge carrier injecting layer for injecting negative charge carriers; and means to enable an electric field to be applied across the said layers wherein at least a part of each of the first and second layers is located in an emission zone of the device, said emission zone extending over a capture region of the device wherein positive and negative charge carriers combine with one another to form excitons and having a width characteristic of the distance over which said excitons migrate before decaying radiatively, such that on application of an electric field to the device both of said first and second polymer layers are caused to emit radiation at their respective wavelengths.

As mentioned above, it is not readily apparent from the work which has already been published in relation to conjugated polymers that it is possible to use a plurality of layers to control the colour of emitted radiation.

The inventors have found that it is possible to define an emission zone in which light is emitted through the radiative decay of excitons. This zone has a width resulting from the

capture region of the device where excitons are formed and which is also related to the diffusion characteristic of the excitons. The width of the emission zone can be approximately the same as that of the captive region or it can extend beyond it where the excitons diffuse from it before decaying radiatively. Thus, by ensuring that both the first and second layers have parts lying in this characteristic width, excitons will be present in both the layers and cause emission of radiation from the first and second layers. The effect is quite clearly ascertainable from the experiments discussed herein but there are different theories which could be developed to support the practical observations. One theory discussed herein is that the characteristic diffusion length for an exciton determines the critical width of the emission zone but there are other possibilities. The inventors have thus determined that it is possible to control the properties of a multilayer electroluminescent device by selecting the thicknesses of the polymer layers so that at least two layers have parts in the emission zone. In one aspect, the invention involves the use of several polymer layers with different band gaps, with layer thicknesses selected to be smaller than or comparable to, the width of the emission zone. This results in excitons in two or more layers, and thus to light emission from the two or more layers. This then gives light emission with a broader spectral range than can be achieved with one layer. This (together with a colour filter, if necessary) may allow fabrication of a white light source.

Whatever the theory underlying the observed effect, the experimental evidence is to the effect that the width of the emission zone is of the order of 50nm. Thus, in the preferred embodiment, the first layer has a thickness which is not greater than 50nm. Of course, more than two such layers can be provided depending on the required colour of emitted radiation. Generally speaking the emission zone will extend for a width not greater than 200nm, but this depends on the nature of the polymer layers and charge carrier injection layers.

The location of the emission zone with respect to the charge carrier injection layers depends on the mobilities of electrons and holes within the polymer layers and on the injection functions and can be determined for each particular case using the models discussed later.

In one embodiment there is a third layer of a semiconductive conjugated polymer between said second polymer layer and said second charge carrier injecting layer, the thickness of the third polymer layer being not greater than 50 nm. The emission zone can include part of two layers, part of one layer and a complete other layer or parts of two layers and a complete other layer.

The electroluminescent device can include an additional layer of a conjugated polymer adjacent the second charge carrier injecting layer which is not necessarily electroluminescent but which instead functions as a barrier layer.

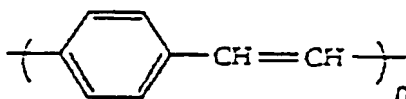
Preferably the second charge carrier injecting layer is calcium and the first charge carrier injecting layer is indium-tin oxide coated onto a glass substrate.

The term "conjugated polymer" used herein indicates a polymer for which the main chain is either fully conjugated, possessing extended pi molecular orbitals along the length of the chain, or else is substantially conjugated, but with interruptions to conjugation at various positions, either random or regular, along the main chain. It includes within its scope homopolymers and copolymers. The present invention can utilise any conjugated polymer which is capable of forming a thin electroluminescent film.

Particularly preferred conjugated polymers include poly(p-phenylene vinylene)PPV and copolymers including that polymer. Preferred features of the polymers used with the respective layers are that they should be stable to oxygen, moisture and to exposure to elevated temperatures, they should

have good adhesion to an underlying layer, good resistance to thermally-induced and stress-induced cracking, good resistance to shrinkage, swelling, re-crystallisation or other morphological changes. Moreover, the polymer film should be resilient to ion/atomic migration processes, for example by virtue of a high crystallinity and high melting temperature. Particularly preferred polymers are discussed in the literature referred to above, particularly in PCT/WO90/13148 the contents of which are herein incorporated by reference. A particularly suitable polymer is a poly(2,5-dialkoxyphenylenevinylene). Examples are MEHPPV, poly(2-methoxy-5-(2-methylpentyloxy)-1,4-phenylenevinylene), poly(2-methoxy-5-pentyloxy-1,4-phenylenevinylene), and poly(2-methoxy-5-dodecyloxy-1,4-phenylenevinylene), or other poly(2,5-dialkoxyphenylenevinylenes) with at least one of the alkoxy groups being a long solubilising alkoxy group, linear or branched. Other suitable conjugated polymers can also be selected from the poly(alkylthienylene)s. One example is poly(3-dodecylthienylene).

The film of conjugated polymer is preferably a film of a poly(p-phenylenevinylene) [PPV] of formula

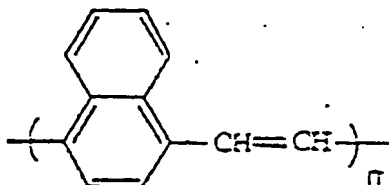


wherein the phenylene ring may optionally carry one or more substituents each independently selected from alkyl (preferably methyl), alkoxy (preferably methoxy or ethoxy) or any other substituent which maintains electroluminescent properties in the conjugated polymer.

Any poly(arylenevinylene) including substituted derivatives thereof or any poly(arylene) is also suitable. Throughout this specification the term "arylene" is intended to include in its scope all types of arylenes including heteroarylenes as well as arylenes incorporating more than one ring structure including fused ring structures.

Other conjugated polymers derived from poly(p-phenylenevinylene) are also suitable for use as the polymer film in the EL devices of the present invention. Typical examples of such derivatives are polymers derived by:

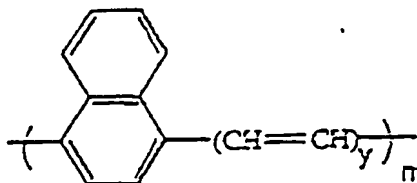
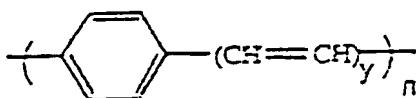
(i) replacing the phenylene ring in formula (I) with a fused ring system, e.g. replacing the phenylene ring with an anthracene or naphthalene ring system to give a structure such as:-



or alternative linkage arrangements which maintain the electroluminescent properties in the conjugated polymer.

These alternative ring systems may also carry one or more substituents of the type described above in relation to the phenylene ring.

(ii) increasing the number of vinylene moieties associated with each phenylene ring (or each of the other alternative ring systems described above in (i) to give structures such as:-



or alternative linkage arrangements which maintain the electroluminescent properties in the conjugated polymer

where y represents 2, 3, 4, 5, 6, 7,.....

Once again, the ring systems may carry the various substituents described above.

These various different PPV derivatives will possess different semiconductor energy gaps. The ordering of the layers within the electroluminescent device is affected by the individual bandgaps. The layers should be ordered so that there is no charge trapping at the boundaries between layers.

Other polymers which are particularly suitable for use in the present invention are the semiconductive copolymers discussed in PCT/WO92/03490, the contents of which are herein incorporated by reference. In a preferred embodiment, the copolymer is a conjugated poly(arylenevinylene) copolymer with a proportion of the vinylic groups of the copolymer saturated by the inclusion of a modified group substantially stable to elimination during formation of a film of the copolymer. The proportion of saturated vinylic groups controls the extent of conjugation and thus modulates the semiconductor bandgap of the copolymer.

Preferably polymers for use in the present invention are capable of being processed either as precursors which are subsequently converted to a conjugated form or as intrinsically soluble polymers. In this regard reference is made to PCT/WO90/13148, the contents of which are herein incorporated by reference.

The invention also provides a method of making an electroluminescent device comprising the following steps: providing a first charge carrier injecting layer for injecting positive charge carriers; depositing on said charge carrier injecting layer a first layer of a soluble polymer in a solution of a first solvent and to a first predetermined thickness; depositing a second layer of polymer in the form of a precursor in a solution of a second solvent to a second

predetermined thickness; heat treating the device so that the precursor is converted to its polymer which is insoluble; and depositing a second charge carrier injecting layer for injecting negative charge carriers, wherein the first and second predetermined thicknesses are selected so that at least a part of each of the first and second layers is located in an emission zone of the device.

Reference is made to our copending application filed on even date entitled "Manufacture of Electroluminescent Devices" (Page White & Farrer Ref. 74148/VRD), the content of which are incorporated herein by reference.

For a better understanding of the present invention and to show how the same may be carried into effect, reference will now be made by way of example to the accompanying drawings.

Brief description of the drawings

Figures 1a, 1b and 1c show the chemical structures of a) PPV, b) a copolymer of PPV and PDMeOPV, c) MEHPPV;

Figure 2a shows the current density against electric field characteristics of each of four devices on a log-log scale;

Figure 2b shows current density against voltage on a linear scale;

Figures 3a to 3d show diagrammatically the structures of four electroluminescent devices I to IV; and

Figure 4 shows the electroluminescent emission spectra (all normalised to a peak emission of 1 and offset) of (a) sample I, (b) sample II, (c) sample III, (d) sample IV, (e) a unilayer copolymer electroluminescent device, (f) a unilayer PPV electroluminescent device, (g) to (i) the absorption spectra of the polymers a, b, and c themselves. Curve g corresponds to polymer c) MEHPPV; curve h corresponds to polymer a) PPV and curve i corresponds to copolymer b).

Description of the preferred embodiment

Three different semiconducting poly(arylenevinylene)s are used to demonstrate the invention.

(a) (Figure 1a) Poly(p-phenylenevinylene), PPV, was processed from a tetrahydrothiophenium (THT)-leaving precursor polymer which is soluble in methanol, PPV has a $\pi - \pi^*$ band gap of about 2.5eV.

(b) (Figure 1b) A copolymer was prepared from a statistical precursor copolymer to PPV and poly(2,5-dimethoxy-p-phenylenevinylene), PDMeOPV, which is soluble in methanol. The monomer feed ratio was 9:1. The synthesis of such a copolymer is described for example in a paper entitled "LEDs based on Conjugated Polymers: Control of Colour and Efficiency by P. Burn, et al given at MRS Boston 1991 and published in Mat. Res. Soc. Symp. 1992 247, 647-654. Under the experimental conditions used here, the band gap of the polymer obtained after thermal conversion is blue-shifted with respect to PPV due to the presence of non-eliminated methoxy groups at the vinylic carbons adjacent to the dialkoxy-substituted phenylenes. The resulting copolymer has a $\pi - \pi^*$ band gap of about 2.6eV.

(c) (Figure 1c) The third polymer used was poly(2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylenevinylene), MEHPPV. Due to the long alkyl side-groups this derivative of PPV is soluble in and processed from chloroform. It has a $\pi - \pi^*$ band gap of about 2.2eV.

Both the THT-leaving group precursors to PPV and the copolymer are such they can be laid down by spin coating in solution of a solvent, which when dry, forms a stable layer onto which a further layer may be put down. This enables the construction of multilayer structures since, once a layer has dried, subsequent deposition of additional polymer layers will not remove the initial layer. The two THT-leaving group precursor polymers are insoluble in chloroform, but soluble in methanol. MEHPPV is soluble in chloroform, but insoluble in methanol. This difference in solvents allows a layer of

precursor to be spin-coated on top of a layer of MEHPPV without removal of the MEHPPV and vice-versa. Hence multilayer structures composed of the three different polymers were fabricated.

The multilayer devices of conjugated polymers were constructed as follows. Indium-tin oxide (ITO)-coated glass substrates were thoroughly cleaned with acetone and subsequently with propan-2-ol, both in an ultrasonic bath. Multilayer structures were formed by spin-coating layers of polymer or precursor, one on top of another as discussed above. All layers were spin-coated within a nitrogen-filled glovebox (O_2 and H_2O content 10ppm), in which all subsequent processing steps were also performed. Film thicknesses of the polymer layers were set by control of both spin-speed and solution concentration as follows: the copolymer at 20nm, MEHPPV at 50nm and PPV at more than 50nm. The thicknesses of the individual polymer layers and total polymer layer were measured with a Dektak IIA surface profiler. The samples were thermally converted at 200°C in vacuo (10^{-6} torr) for 12 hours to convert the precursor polymers. Calcium contacts were vacuum deposited on the samples and the samples were hermetically sealed. Sample areas were 1 mm². Four multilayer device structures have been studied here; details of construction are summarised in table 1 and illustrated in Figures 3a to 3d.

To form device I illustrated in Figure 3a, an indium-tin oxide coated glass substrate 1 was spin coated firstly with a precursor to the copolymer (b) at a thickness of 20nm (layer 21). The layer was allowed to dry and then a precursor to PPV (a) in the thickness of 230nm (layer 22) was laid down by spin coating, and allowed to dry. Finally a layer 23 of MEHPPV (c) in the thickness of 50nm was laid down by spin coating. Layers 21 and 22 were put down in a solution of methanol and layer 23 was put down in a solution of chloroform. The sample was then heat treated to cause thermal conversion of the precursors to the copolymer in layer 21 and to PPV in layer

22. Finally a calcium contact 2 was vacuum deposited on layer 23.

Device II as illustrated in Figure 3b was formed as follows. Onto an indium-tin oxide coated glass substrate 1 there was spin coated a first layer 31 of MEHPPV in the thickness of 50nm which was allowed to dry, and then a precursor to PPV 32 in the thickness of 50nm and allowed to dry. Then a precursor to the copolymer 33 was laid down by spin coating in the thickness of 20nm. The first layer 31 was put down in a solution of chloroform and the second and third layers 32,33 were put down in a solution of methanol. The device was heat treated to thermally convert the precursors in layers 32 and 33 to the respective polymers PPV and the copolymer and a calcium contact 2 was then vacuum deposited onto layer 33.

Device III illustrated in Figure 3c was formed as follows. Onto an indium-tin oxide coated glass substrate 1 was spin coated a first layer 41 of a precursor to the copolymer in a thickness of 20nm. The precursor was allowed to dry. A second layer 42 of MEHPPV is laid down by spin coating in the thickness of 50nm and allowed to dry and then a third layer 43 of the precursor to PPV is laid down by spin coating in a thickness of 150nm. After this layer had dried a fourth layer 44 of a precursor to the copolymer was laid down by spin coating in a thickness of 20nm. The device was then heat treated to thermally convert the precursors to their respective conjugated polymers and a calcium contact 2 was vacuum deposited onto layer 44.

Device IV illustrated in Figure 3d was formed as follows. Onto an indium-tin oxide coated glass substrate 1 was spin coated a precursor 51 to the copolymer in the thickness of 20nm. This was then allowed to dry. A precursor to PPV 52 was laid down by spin coating in a thickness of 30nm and allowed to dry and then a precursor to the copolymer 53 was laid down by spin coating in the thickness of 20nm. The

sample was then heat treated and a calcium contact 2 was vacuum deposited onto the third layer 53.

In these structures, the calcium contact 2 serves as a cathode for injecting negative charge carriers and the indium-tin oxide serves as an anode for injecting positive charge carriers. Reference numeral 4 indicates a negative contact and reference numeral 6 indicates a positive contact.

In devices II, III and IV the layer adjacent the calcium contact 2 is a layer of the copolymer which, after heat treatment, is insoluble in common solvents. This layer acts as a barrier layer to shield the cathode from chemical attack, while allowing the injection of charge carrier into the emissive region. This layer of copolymer also serves to resist the diffusion of mobile ions released from the reactive cathode through the polymer layer. It also provides the device with strength, mechanical rigidity and stability at high temperatures. Furthermore, it protects the soluble polymer (MEHPPV) so that the desirable properties of MEHPPV such as the required colour or high efficiency for luminescence can be exploited. Other conjugated polymers or other polymers which can be used as barrier layers instead of that illustrated in Figure 1b are methoxy-substituted precursor polymers to PPV, dimethoxy-PPV, dimethyl-PPV, polyaniline and derivatives, polyvinylcarbazole, blends of dimethyl-PPV and any other higher band gap polymers.

As explained above, the indium-tin oxide coated glass substrate serves as an anode for the device while the calcium contact 2 serves as a cathode. These constitute means for enabling an electric field to be applied across the layers.

The devices were driven by applying a positive bias to the ITO with a Keithley 230 voltage source and electric currents were monitored with a Keithley 195A multimeter.

Electroluminescence (EL) was observed through the ITO substrate. Integrated light output was monitored with a

large-area calibrated silicon photodiode in photoamperic mode, measuring the photocurrent as a voltage across a $1\text{M}\Omega$ resistor. Spectrally-resolved EL measurements were made using a Coderg PH1 double monochromator and a Hamamatsu R943-02 photomultiplier tube. Internal quantum efficiencies, defined here as photons generated per injected charge, were measured at a device brightness of about 5cd/m^2 . In calculating the internal quantum efficiency account has been taken of the effects of refraction by the glass substrate, as discussed in an article in the 1992 issue of Synthetic Metals authored by Brown, et al. UV/vis absorption spectra were recorded with a Perkin Elmer $\lambda 9$ spectrophotometer on samples of polymer spin-coated on silica substrates.

The four device configurations show colours of emission from red to yellow/green. Electrical and electro-optical characteristics for these devices are shown in Table 1, and in Figures 2a, 2b and 4. In Table V, the layers are denoted in numerical order from the ITO content 1. In Figures 2a and 2b note that different thicknesses of the combined polymer layers give different drive voltages, but that these scale to give a universal curve for current density versus electric field. Figure 2b shows the current density against electric field voltage characteristics of the four devices. Figure 4 shows the emission spectra from the various devices (the spectra are offset and normalised to give equal peak heights at the peak emission wavelengths). Also displayed are emission spectra from unilayer devices of PPV and the copolymer. The absorption spectra of the 3 polymers are also shown (curves g,h,i) (normalised to give a peak $\pi - \pi^*$ absorption coefficient of 1), showing the differing optical band gaps.

Figures 2a and 2b show clearly that the current density under forward bias is determined primarily by the electric field (calculated as the applied voltage divided by the sum of the individual layer thicknesses). In contrast to the results obtained for the devices formed with PPV and the butyl PBD/PMMA layers (discussed in the reference referred to above

by P.L. Burn et al), the devices studied here do not show a large build-up of space charge at the interfaces between polymer layers. Also, the small differences in band gap for the polymers in contact with the metal electrode layers do not affect significantly the threshold fields for charge injection.

The various structures give a range of different emission colours, and by analysing these it can be identified which of the polymer layers are acting as sites for radiative decay of excitons:

Structure I was the only structure to show an emission spectrum characteristic of a single polymer layer, in this case MEHPPV. There is no contribution to the emission spectrum from excitons decaying in the PPV or copolymer layers, since at energies about 2.35eV there is no emission, whereas the emission spectra of PPV and copolymer both have strong emission intensities at 2.35eV. Probably therefore electron injection is the limiting process in the devices, so that electron and holes meet in a recombination zone adjacent to the electron-injecting calcium contact 2. It is unfavourable for the excitons formed in the MEHPPV layer to migrate into the other polymer layers which have larger band gaps. By the same argument, the absence of any contribution to the emission spectra from the PPV layer indicates that no electron-hole capture occurs in this layer and thus that no electrons migrate into the PPV layer through the MEHPPV layer, under the influence of the electric field. The copolymer and PPV layers are acting as hole transport layers. The emission spectra also demonstrate that, surprisingly, the thermal conversion process does not damage or alter the MEHPPV.

Structure II has the same combination of polymer layers as structure I but constructed the other way up. The emission spectrum of the device is broad with emission stretching from below 1.55eV to above 2.5eV. At 1.75eV, both PPV and copolymer have negligible emission yet the emission of sample II is still more than 15% of the peak emission and must be due to the radiative decay of excitons located in the MEHPPV

layer. Similarly, at 2.35eV MEHPPV has zero emission, yet the emission of sample II is still more than 30% of the peak emission for the sample and so the emission at this energy must be due to the decay of excitons located in either the copolymer or PPV layers. This device is thus clearly an example of a conjugated polymer device emitting from more than one polymer layer. The nature of the generation of the excitons in the various polymer layers will be discussed later.

Structure III has an emission spectrum very similar to that for structure II. Thus the emission of this device, for the same reasons as discussed above, has contributions from the decay of excitons located in the MEHPPV layer and at least one of either the PPV or the copolymer layers. It appears the additional copolymer layer has negligible effect on the emission spectrum, and one might deduce that no excitons indeed are located in this additional copolymer layer adjacent to the ITO. This device also is an example where exciton decay occurs within the bulk of a polymer multilayer device as the MEHPPV is separated from both contacts by other layers.

Structure IV has an emission spectrum with contributions from both the PPV layer and the copolymer layer. The emission spectrum shows phonon structure at 2.07, 2.26 and 2.39eV which is less pronounced than that for PPV, though clearer than in the copolymer.

These devices operate by electron and hole injection at negative and positive electrodes, with electron-hole capture to form excitons, which can then decay radiatively. There are no large changes in the current density-electric field characteristics accompanying the introduction of several layers of polymer, so there is probably little or no charge confinement resulting in space charge build-up at the interfaces between these layers. Thus there is little modification of the internal electric field and the conditions for charge injection and charge migration are not much

affected by the presence of several layers of polymer with similar band gaps.

In structures II-IV emission in several polymer layers can be observed with an emission zone more than 50nm in total thickness. It is of interest to identify the mobile species which gives rise to this wide emission zone. The two candidate mobile species are (i) charge carriers and (ii) neutral excitons. Depending on the behaviour of these charged and neutral species, we can expect to see behaviour ranging from the limit where a broad electron-hole capture zone is narrow, but in which exciton diffusion acts to produce a broad emission zone.

Thus, the precise thicknesses and arrangement of the layers of conjugated polymer within an electroluminescent device can be ascertained from a knowledge of the polymer band gap, electron-hole mobilities in the polymer, and a value for the exciton diffusion lifetime with reference to the models discussed herein. Firstly these cases are outlined by two simple models to give estimates of the widths produced by the two mechanisms.

(i) Width of electron-hole capture zone.

Let us consider that electron hole capture is mediated by Coulombic attraction and will occur within a sphere of radius, r_{capture} , where this attraction exceeds the thermal energy. Then

$$kT = \frac{e^2}{4\pi\epsilon_0\epsilon_r r_{\text{capture}}}$$

$$\therefore r_{\text{capture}} \leq \frac{e^2}{4\pi\epsilon_0\epsilon_r kT}$$

Taking a value for the relative permittivity ϵ_r of about 4, we have at room temperature $r_{\text{capture}} \approx 14\text{nm}$. It can be demonstrated from simple models for the drift of charges through the device that, in the absence of barriers for carrier transport at interfaces, the space charge densities of positive and negative charge carriers are low, and that the width of the electron-hole capture zone can be modelled to lie in the range of the polymer layer thicknesses used for example II to IV. At an electric field, F , with a current density, j , and a carrier mobility, μ , the carrier density, p is given by

$$p = j/ev = j/eF\mu$$

Taking typical device operation values of $j = 1 \text{ mA/cm}^2$, and $F = 10^8 \text{ V/m}$ and assuming a mobility, $\mu = 10^{-4} \text{ cm}^2/\text{V sec}$, as has been observed in similar conjugated polymers for the majority, p-type carriers gives a carrier density, $p = 6.2 \times 10^{20} \text{ m}^{-3}$. This gives a carrier-carrier separation of about 120 nm.

If electrons have a mobility, μ_n , the number of collisions an electron undergoes in time, t , is $tF(\mu_n + \mu_p)p\sigma$ where σ is the collision cross-section. Assuming every collision results in capture, we can then estimate an electron-hole capture zone width, W_{e-h} given by

$$W_{e-h} = \frac{\mu_n}{(\mu_n + \mu_p)p\sigma} = \frac{\mu_n \mu_p e F}{(\mu_n + \mu_p)j\sigma} = \frac{\mu_n}{(\mu_n + \mu_p)p r_{\text{capture}}^2}$$

Using the values here, based on the hole mobilities estimated above, and assuming equal electron and hole mobilities, we find a value of $W_{e-h} \approx 4000 \text{ nm}$. This width is very much larger than we find here, and we consider that confinement to much smaller thicknesses near the cathode results from a much lower electron mobility.

(ii) Exciton diffusion length.

We may assume a simple exciton diffusion equation, with a characteristic variance of the displacement from the origin, $l^2 = D\tau$. Exciton lifetimes, τ , of above 0.25 ns have been measured in PPV. Typical values of the magnitude of diffusion coefficients, D , in molecular semiconductors are of the order $1 \times 10^{-3} \text{ cm}^2 \text{ s}^{-1}$ and the coefficient in these conjugated polymers does not exceed this. These values then give an estimate of the diffusion length of an exciton, l , ~5 nm.

Although the multilayer emission is observed only in structures II-IV, it is constructive to consider an analysis of structure I first. As the emission spectrum of structure I is characteristic of the single polymer layer of MEHPPV, the recombination zone is probably confined within the 50 nm thickness of the MEHPPV layer. From the above analysis, such a zone thickness would only be consistent with electron-hole capture by a Coulomb interaction if the electron mobility is less than $10^{-6} \text{ cm}^2/\text{V sec}$. This may be the case, since minority carrier mobilities in organic semiconductors are often significantly smaller than those of the majority carriers. Another explanation is that there is a barrier against electron migration from the MEHPPV layer into the PPV layer and so confining the recombination zone to the MEHPPV layer.

As has already been indicated, in structures II-IV the spectral shifts indicate that emission occurs in layers not solely adjacent to the calcium contact. In the scenario in which the width of the emissive layer is determined by the width of the electron-hole capture zone, there is no barrier against electron migration in all these structures since electrons pass from PPV into MEHPPV. The electron-hole capture zone then extends through several polymer layers and over more than 200 nm in structure III. Excitons are then formed in several different polymer layers and then radiatively decay in several polymer layers, given the

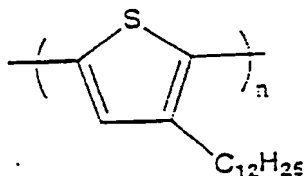
observed emission spectra. Thus, in structure I the recombination zone is within 50 nm of the electron-injecting contact, observing emission from only one layer. The recombination zone is probably limited in this structure due to a barrier against electron migration out of the MEHPPV layer.

Though the inventors consider that the width of the characteristic emissive zone in these EL devices is in part determined by the statistics for electron-hole capture, there are other possible explanations. One such is that the recombination could be occurring within a 50 nm zone adjacent to the calcium contact (as in structure I) and that there is subsequent exciton transfer from the recombination zone to different emission zones. This transfer could occur by several mechanisms. Firstly, there may be absorption and re-emission at a longer wavelength, but this is not significant for the devices studied here. One reason for this is that one would expect to see a concomitant fall-off in device efficiency, given that luminescence yields are not 100%. Experimentally, there is little variation in efficiency from one device to another, as summarised in table 1.

Secondly, there may be exciton diffusion from one layer to another. The radiative lifetimes for singlet excitons in PPV are of the order of 1 nsec. Measured photoluminescence lifetimes are lower, indicating that there are competing non-radiative decay mechanisms, but lifetimes are 250 psecs or longer at room temperature, and this allows for diffusion of the exciton through the polymer prior to decay. Evidence for migration of excitons, created by photoexcitation, within a film of PPV, is provided by comparison of absorption and photoluminescence spectra. Even in polymer samples that show a considerable spread in intra-chain conjugation length, and hence band gap, such that the phonon structure expected in the absorption spectrum is washed out, the luminescence spectra show clear vibronic structure, indicating that the excitons so formed, prior to emission, have migrated to the chain segments

with longest uninterrupted conjugation length and hence with lower band gap. Exciton diffusion is very well studied in molecular semiconductors and under the conditions in these conjugated polymer films may occur either by direct hopping or by Förster transfer. Energy transfer to a chromophore with a longer wavelength of emission, which can be selectively introduced as a dopant in the semiconductor, is a well-studied phenomenon.

A further example of the invention is discussed below. A different soluble conjugated polymer is used in place of the MEH-PPV. The polymer used is poly(3-dodecylthienylene), P3DT, for which the chemical structure is



This polymer is soluble by virtue of the dodecyl side-chain, and is processed from solution, in for example, chloroform. It shows good photoluminescence and has been shown to function in an electroluminescent device.

A device was fabricated in a similar manner to Example II, with a layer of P3DT (thickness 500 nm) on the ITO contact 1, and a layer of PPV precursor spin-coated on top to a thickness of 100nm and subsequently heated to convert to PPV (200°C, 12 hours) in place of the two precursor-route layers in Example II. Calcium was evaporated onto this as a final stage.

While these particular thicknesses were found to be successful, it is desirable to have a thickness of less than 500nm for P3DT. This is acceptable provided that at least part of both of the layers lie in the emission zone of the device.

This device shows a high threshold voltage for charge injection, requiring 80 V or more to give readily visible

emission. This high drive voltage is due to the considerable thickness of the P3DT layer. The quantum efficiency for this device was found to be about 0.08%.

The light output was observed by eye to be deep red, characteristic of the luminescence of P3DT which is considerably to the red of PPV. Emission from the PPV layer viewed through the P3DT layer and the ITO/glass substrate would be strongly attenuated by the absorption of the PPV emission by the P3DT (the PPV emission falls in the absorption band of P3DT since this has a lower band gap).

This device demonstrates various points:

- (i) Emission is produced in a layer not immediately adjacent to the cathode. It can reasonably be expected that emission was also observed from the PPV layer, (c.f Example II). This is therefore another example of a multilayer emitting device, but should optimally be modified so that the polymer layer thicknesses avoid the problem of absorption by one layer of the light produced by another.
- (ii) Furthermore, it is a device where a layer of precursor polymer has successfully been laid down onto a soluble polymer layer, taking advantage of the different solubilities of the polymers, and where the structure survives the thermal treatment required to convert to the conjugated form.

Industrial Application

In summary, the inventors have discovered that multilayer electroluminescent devices with emission from more than one layer can be fabricated. Such multilayer structures could be used to control device characteristics in a range of applications. For example at least two polymer layers can be arranged to be simultaneously luminescing. This will allow generation of a broader spectrum than can be achieved with a

single layer (see structures II, III and IV), and may allow fabrication of a white-light source.

A particularly important discovery is that the region of the device in which the emission occurs can be arranged to be sufficiently wide so that several polymer layers can simultaneously emit. The probable reasons for this are firstly, that in the absence of charge trapping at an interface between two polymer layers, the electron-hole capture process requires thicknesses of polymer of at least 50 nanometers so that excitons are formed in the region of this thickness. Secondly, excitons once formed will diffuse before they decay and this process of diffusion can further broaden the width of the emission zone. The inventors have determined that for optimum performance the device should be manufactured so as to take into account the following points. As is evident from the examples, the emission is usually to be seen from the region of the polymer adjacent the cathode. This indicates that either electron injection is more difficult than hole injection and/or that electrons have lower mobility than holes. Emission from more than one layer is therefore more likely to be achieved in devices where appropriate layers are deposited close to the cathode. Another determinative feature is the band gap of the polymer layers. Inappropriate ordering of the polymer layers can result in complete trapping of excitons within one layer only as seen in Example 1 in which there is a low band gap polymer (MEHPPV) adjacent the cathode. Thus, preferably, the band gaps are ordered to assist charge carrier transfer and capture.

Sample Number	I	II	III	IV
Layer 1	copolymer	MEHPPV	copolymer	copolymer
thickness (nm)	20	50	20	20
Layer 2	PPV	PPV	MEHPPV	PPV
thickness (nm)	230	50	50	30
Layer 3	MEHPPV	copolymer	PPV	copolymer
thickness (nm)	50	20	150	20
Layer 4			copolymer	
thickness (nm)			20	
Colour of light emission	red	orange	orange	yellow
Quantum efficiency, η (%)	0.30	0.22	0.24	0.16
Electric field (V/cm) required to produce:				
1 mA cm ⁻²	8.0×10^5	7.1×10^5	7.4×10^5	7.1×10^5
2 mA cm ⁻²	8.6×10^5	7.7×10^5	7.9×10^5	7.7×10^5

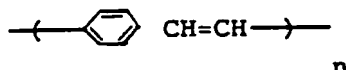
Table 1. Characteristics of the four multilayer electroluminescent devices. Polymer layer 1 was spin-coated directly on top of the ITO contact. The calcium contact was evaporated on top of polymer layer 3 (or polymer layer 4 in the case of device III).

CLAIMS:

1. An electroluminescent device comprising:
 - a first charge carrier injecting layer for injecting positive charge carriers;
 - a first layer of a semiconductive conjugated polymer having a band gap selected such that when it is excited radiation at a first wavelength is emitted;
 - a second layer of a semiconductive conjugated polymer having a band gap selected such that when it is excited radiation at a second wavelength is emitted;
 - a second charge carrier injecting layer for injecting negative charge carriers; andmeans to enable an electric field to be applied across the said layers wherein at least a part of each of the first and second layers is located in an emission zone of the device, said emission zone extending over a capture region of the device wherein positive and negative charge carriers combine with one another to form excitons and having a width characteristic of the distance over which said excitons migrate before decaying radiatively, such that on application of an electric field to the device both of said first and second polymer layers are caused to emit radiation at their respective wavelengths.
2. An electroluminescent device as claimed in claim 1 wherein the width of the emission zone is not greater than 200nm.
3. An electroluminescent device as claimed in claim 1 or 2 wherein the first layer has a thickness which is not greater than 50nm.
4. An electroluminescent device as claimed in claim 1 or 2 wherein there is a third layer of a semiconductive conjugated polymer between said second polymer layer and said second charge carrier injecting layer.

5. An electroluminescent device as claimed in claim 4 wherein the third layer has a thickness not greater than 50nm.
6. An electroluminescent device as claimed in claim 4 or 5 wherein the third layer constitutes a barrier layer adjacent the second charge carrier injecting layer.
7. An electroluminescent device as claimed in claim 4, 5 or 6 wherein the third layer is light emissive.
8. An electroluminescent device as claimed in any preceding claim wherein there is a further polymer layer between the first layer and the first charge carrier injecting electrodes.
9. An electroluminescent device as claimed in any preceding claim wherein the second charge carrier injecting layer is calcium.
10. An electroluminescent device as claimed in any preceding claim wherein the first charge carrier injecting layer is indium tin oxide.
11. An electroluminescent device as claimed in any preceding claim wherein the semiconductive conjugated polymer is selected from the group comprising poly(p-phenylene vinylene); substituted derivatives of poly(p-phenylene vinylene); and copolymers including that polymer.
12. An electroluminescent device as claimed in any of claims 1 to 10 wherein the first polymer layer is a poly(2,5-dialkoxyphenylene-vinylene) selected from the group comprising MEHPPV; poly(2-methoxy-5-(2-methylpentyloxy)-1,4-phenylenevinylene); poly(2-methoxy-5-pentyloxy-1,4-phenylenevinylene); and poly(2-methoxy-5-dodecyloxy-1,4-phenylenevinylene).

13. An electroluminescent device as claimed in any of claims 1 to 12 wherein the second polymer layer is PPV of formula



14. An electroluminescent device as claimed in any of claims 1 to 10 wherein the first polymer layer is selected from the poly(alkylthienylene)s.

15. An electroluminescent device as claimed in claim 11 wherein the copolymer is a conjugated poly(arylene vinylene) copolymer with a proportion of the vinylic groups of the copolymer saturated by the inclusion of a modifier group substantially stable to elimination during formation of a film of the copolymer.

16. An electroluminescent device as claimed in claim 1 wherein the first layer is a layer of MEHPPV having a thickness of about 50nm and the second layer is PPV having a thickness of less than 200nm.

17. An electroluminescent device as claimed in claim 16 which includes a barrier layer between the second layer and the second charge carrier injecting layer having a thickness of about 20nm.

18. An electroluminescent device as claimed in claim 1 wherein the first layer is a layer of P3DT having a thickness of about 500nm and the second layer is PPV having a thickness of about 100nm.

19. An electroluminescent device as claimed in claim 4, 5, 6 or 17 wherein the third layer is a copolymer of PPV and PDMeOPV.

20. A method of making an electroluminescent device comprising the following steps:

providing a first charge carrier injecting layer for injecting positive charge carriers;

depositing on said charge carrier injecting layer a first layer of a soluble polymer in a solution of a first solvent and to a first predetermined thickness;

depositing a second layer of polymer in the form of a precursor in a solution of a second solvent to a second predetermined thickness;

heat treating the device so that the precursor is converted to its polymer which is insoluble; and

depositing a second charge carrier injecting layer for injecting negative charge carriers, wherein the first and second predetermined thicknesses are selected so that at least a part of each of the first and second layers is located in an emission zone of the device.

21. A method according to claim 20 when used to manufacture a device according to any of claims 1 to 19.

Fig. 1c

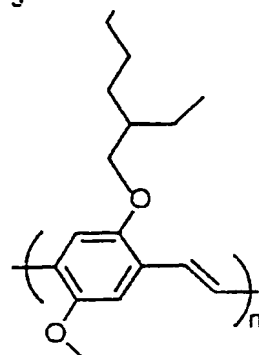


Fig. 1a

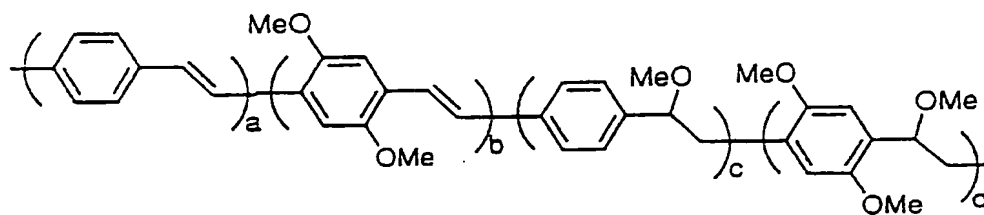
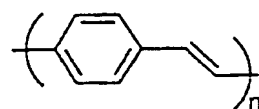
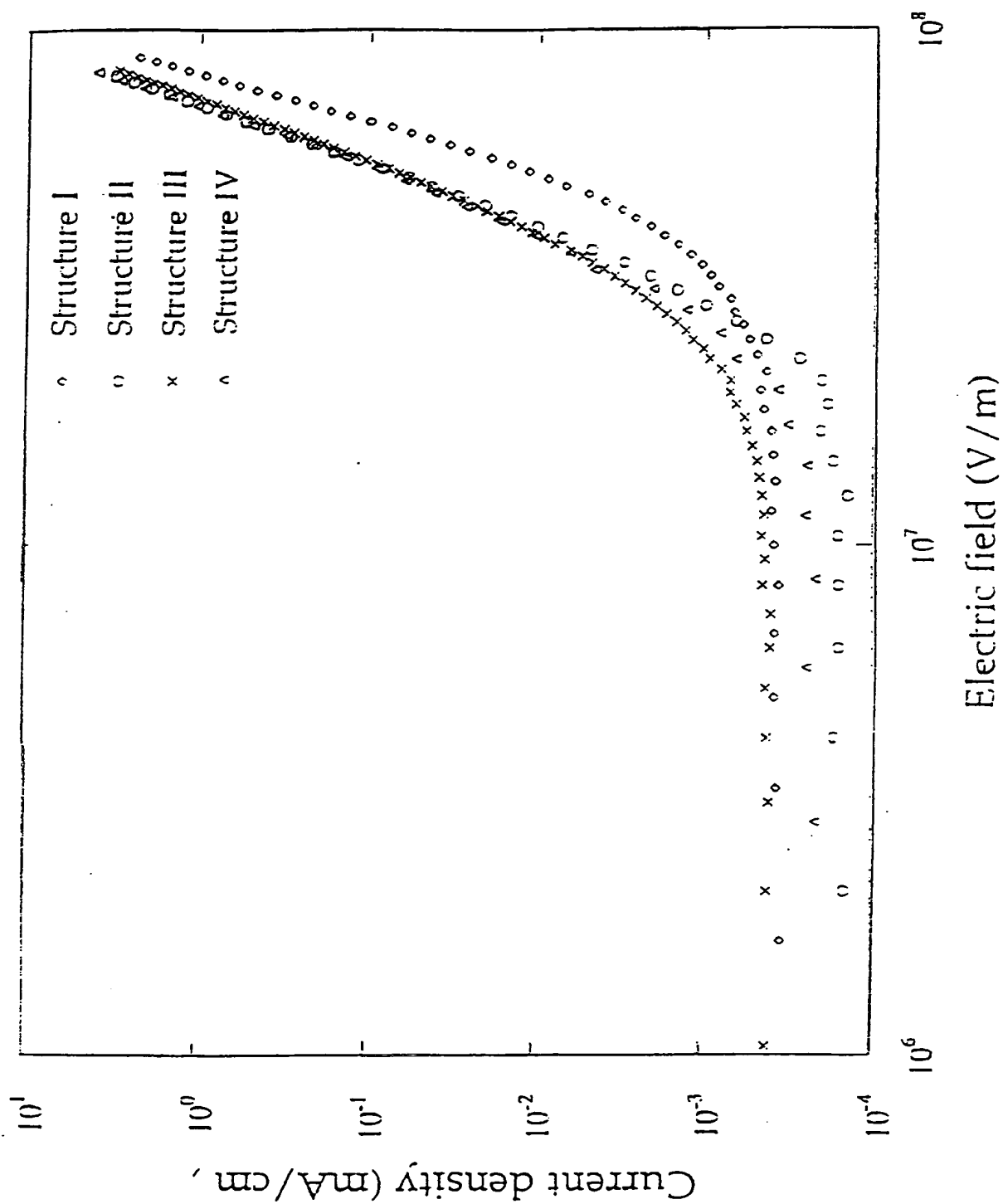


Fig. 1b

Fig. 2a



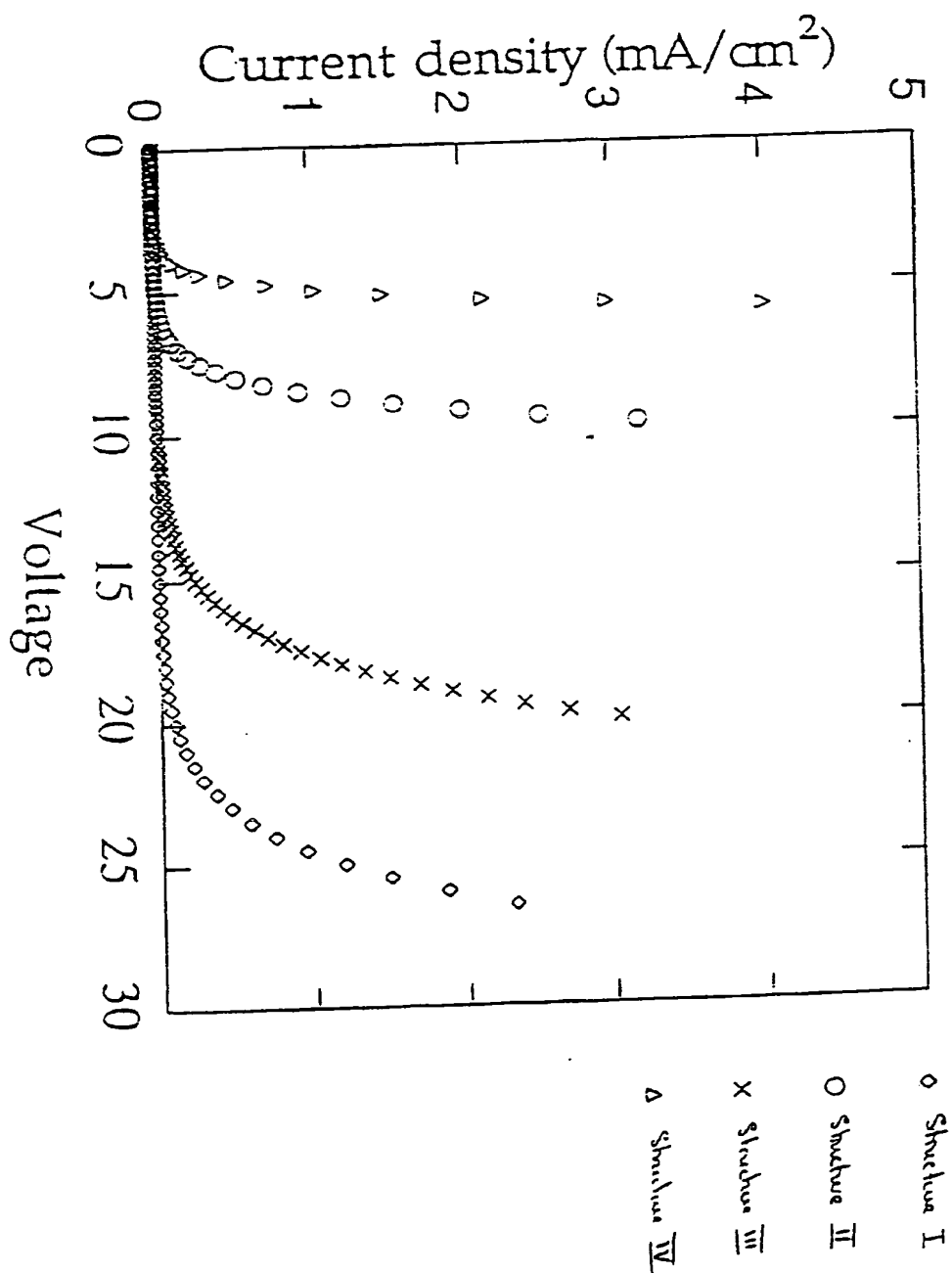


Fig. 2b

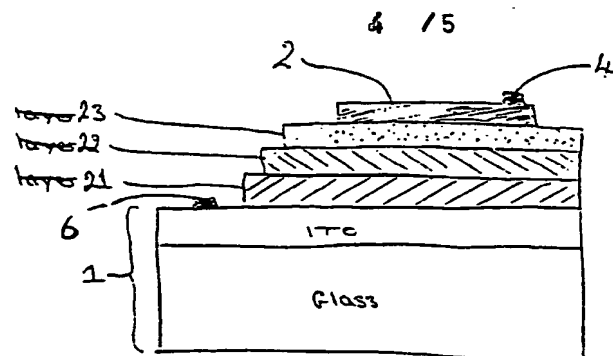


Fig. 3a

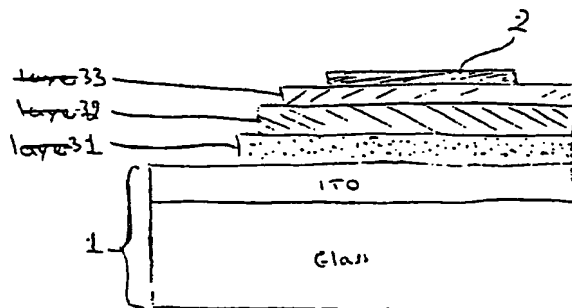


Fig. 3b

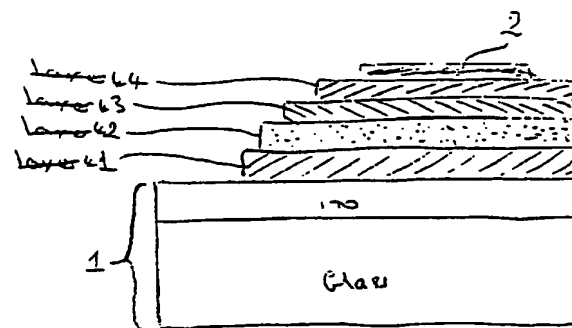


Fig. 3c

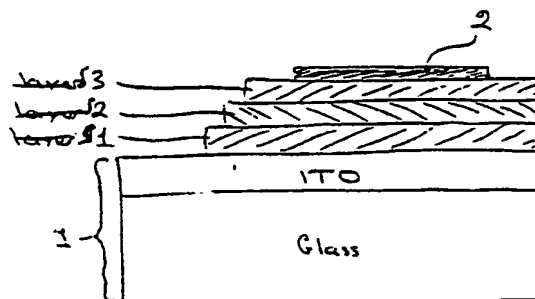


Fig. 3d

Emission spectra
(normalised and displaced)
(arbitrary units)

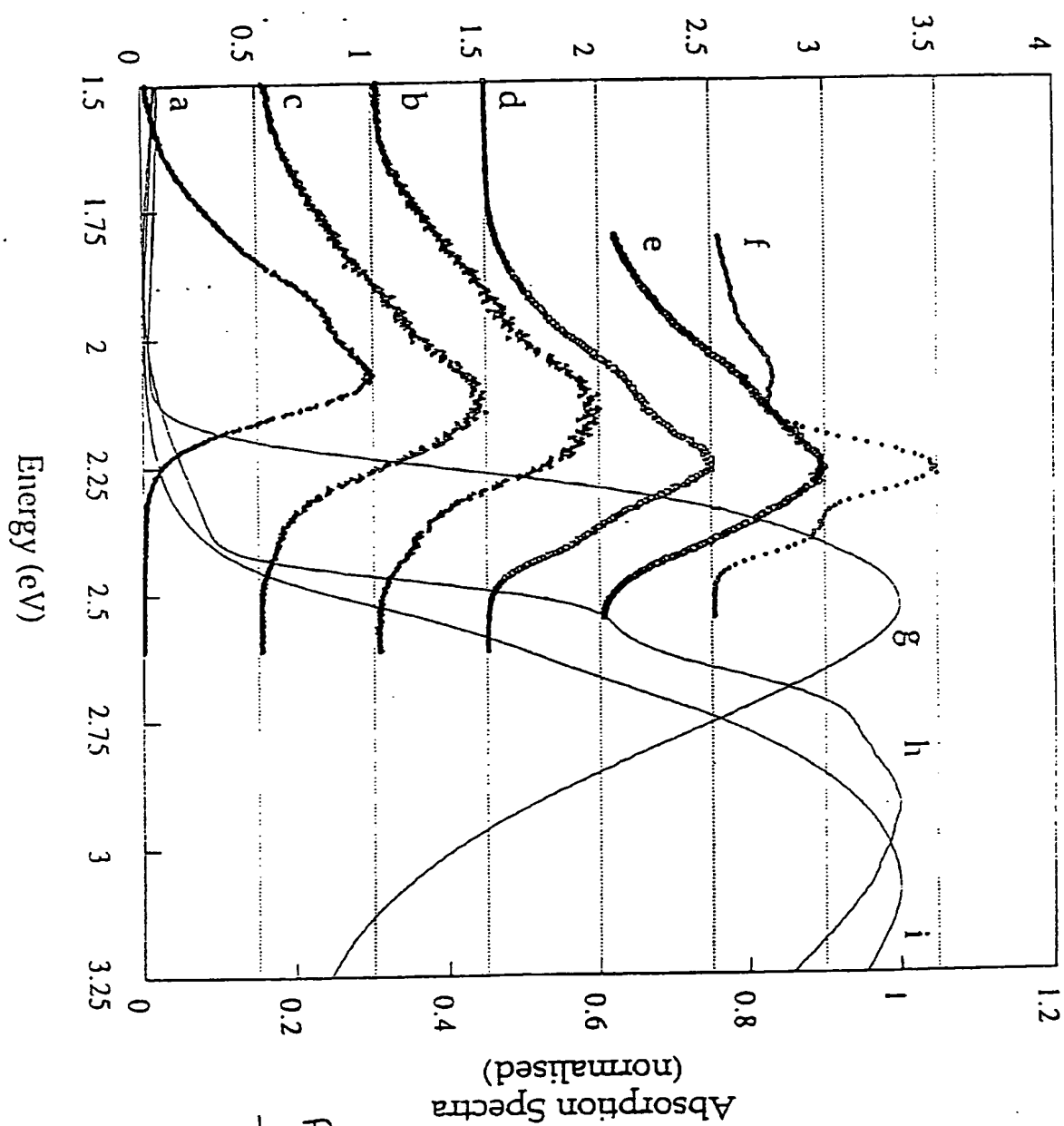


Fig. 4

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 93/01574

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 H05B33/14; H05B33/10; C09K11/06		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	H05B ; C09K	
Documentation Searched other than Minimum Documentation to the extent that such Documents are included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
E	EP,A,0 553 950 (TOSHIBA) 4 August 1993 see claims 1-14; example 36 ---	1-7, 10
A	EP,A,0 443 861 (SUMITOMO) 28 August 1991 cited in the application see claims 1-14 ---	1-3, 11-14
A	EP,A,0 390 551 (TOSHIBA) 3 October 1990 see claims 1-16 ---	1-7, 10
A	WO,A,9 203 491 (CAMBRIDGE RESEARCH) 5 March 1992 see claims 1-28 ---	11-13
		-/--
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IV. CERTIFICATION		
Date of the Actual Completion of the International Search		Date of Mailing of this International Search Report
29 SEPTEMBER 1993		19.10.93
International Searching Authority		Signature of Authorized Officer
EUROPEAN PATENT OFFICE		DROUOT M.C.

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	WO,A,9 203 490 (CAMBRIDGE RESEARCH) 5 March 1992 cited in the application see claims 1-51	11-13,15
A	WO,A,9 013 148 (CAMBRIDGE RESEARCH) 1 November 1990 cited in the application see claims 1-27	1-3, 11-13

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
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GB 9301574
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : A61K 7/09, 7/08	A1	(11) International Publication Number: WO 95/03031 (43) International Publication Date: 2 February 1995 (02.02.95)
(21) International Application Number: PCT/US94/07813 (22) International Filing Date: 19 July 1994 (19.07.94) (30) Priority Data: 093,956 21 July 1993 (21.07.93) US (71) Applicant: AMINCO, INC. [US/US]; P.O. Box 8841, Wilmington, DE 29899 (US). (72) Inventors: COWSAR, Donald, R.; 24 Twelve Oaks Drive, Savannah, GA 31410 (US). ADAIR, Tony, R.; 7 Mulberry Court, Savannah, GA 31406 (US). (74) Agents: LANDSMAN, Sheldon, I. et al.; Sughrue, Mfon, Zinn, MacPeak & Seas, 2100 Pennsylvania Avenue, N.W., Washington, DC 20037-3202 (US).		(81) Designated States: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, ES, FI, GB, GE, HU, JP, KE, KG, KP, KR, KZ, LK, LT, LU, LV, MD, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG), ARIPO patent (KE, MW, SD). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: HAIR RELAXER COMPOSITIONS (57) Abstract <p>The present invention relates to a two-component hair relaxer system comprising (a) a first component comprising a cream base containing a water-soluble salt of a relatively strong base with an anion capable of being precipitated by an alkaline earth metal ion under highly alkaline conditions, and (b) a second, separate component, which is substantially free of water, and containing an alkaline material having an alkaline earth metal ion which forms a precipitate with the anion when the first component and second component are mixed.</p>		

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HAIR RELAXER COMPOSITIONS

FIELD OF THE INVENTION

The present invention relates to a two-component hair relaxer system which produces a highly alkaline hydroxide relaxer upon mixing of the two components, and to a process for making the relaxer. More particularly, the present invention relates to a two-component system in which the concentration of the highly alkaline hydroxide in the relaxer can be accurately controlled when less than a full kit volume is used. Further, the present invention is directed to a hair relaxer system with improved product stability and shelf life.

BACKGROUND OF THE INVENTION

Aqueous highly alkaline hair relaxer (straightener) compositions are known in the art in the form of alkaline, oil-in-water emulsions which derive their chemical activity from either (1) alkali metal hydroxides, (2) quaternary ammonium hydroxides, or (3) guanidine hydroxide dissolved or suspended in the water phase of these hair-treatment compositions such that the pH values of these emulsions are in the range of from 12 to 14 ("highly alkaline"). It is widely and generally accepted that it is the hydroxide ion, which is the alkaline chemical spe-

cies common to all three of the above classes, that is the essential active ingredient in these "strong base" relaxers. It has been proven that hydroxide ions, when inside the cortex of the hair, readily
5 extract acidic alpha protons from the cysteine moieties of hair keratin leading to reversible beta elimination of alkyl disulfide (opening of crosslinks) with the concomitant formation of dehydroalanine. As these crosslinks continuously open and
10 reform, the curly hair is relaxed to a permanently straight configuration.

Although it is the hydroxide anion which is responsible for initiating the chemical reactions within the hair shaft which lead to straightening,
15 it is the cation with which the hydroxide is associated that distinguishes the various known highly alkaline relaxer types from each other. Prior to 1979, one-component ("no-mix") highly alkaline hair relaxer compositions deriving from either sodium or
20 potassium hydroxides were known. In 1979, a mix-type "no-lye" relaxer containing guanidine hydroxide was introduced. A no-lye relaxer is one that does not contain sodium or potassium hydroxide. Because
25 guanidine hydroxide is not stable for long periods in aqueous solutions, it must be prepared fresh just prior to using. Guanidine hydroxide is generally

prepared by mixing an inorganic alkaline earth hydroxide with an aqueous solution of a salt of the strong organic base guanidine, where the anion of the salt is capable of being precipitated by the cation of the alkaline earth hydroxide. In commercially available products of this type, the guanidine hydroxide is generally prepared using calcium hydroxide and guanidine carbonate.

Two-component systems for the preparation and use of guanidine hydroxide relaxers are well documented in the patent literature, as disclosed, for example, in U.S. Patent No. 4,304,244. The first component comprises a viscous cream base containing calcium hydroxide and the second component, known as an activator, comprises an aqueous solution of guanidine carbonate. The two components are combined to form an activated cream containing guanidine hydroxide. U.S. Patent No. 4,304,244 discloses a two-component composition, and states that one or both of the components may be in the form of an aqueous solution, or both components may be in a non-aqueous form with water additionally added to the admixed components. The patent states that when a two-component system is used, the individual components may vary from a solid component to a very dilute solution or dispersion. The patent states

that it is decidedly preferred that the hydroxide component and guanidine salt be packaged as separate components, especially with each component in the form of an aqueous solution, as the resulting mixing of the components is greatly facilitated thereby.

All of the examples which employed a two-component system used a cream base containing an alkaline earth metal hydroxide (e.g., calcium hydroxide) and a guanidine carbonate solution in a weight ratio of at least 70% cream base to 30% guanidine carbonate solution.

The volume of liquid activator in the commercially available two-component systems is typically 50 to 80 ml, and is contained in a container or bottle with a narrow mouth to facilitate pouring.

The volume of the cream base is generally 200 to 220 ml and is contained in a wide-mouth container to facilitate the stirring process when the liquid activator is poured into the cream base. Both the cream base and the liquid activator have a significant amount of water present.

The liquid activator contains a sufficient amount of water to ensure that the guanidine carbonate is completely dissolved in the activator. The guanidine carbonate is typically present in the activator in a high concentration of 20 to 30% by

weight. The cream base, which is an oil-in-water emulsion, typically contains about 40 to 60 weight percent water, and 4 to 7 weight percent calcium hydroxide. The calcium hydroxide generally is present in a molar excess with respect to the guanidine carbonate. The cream base to liquid activator weight ratio is generally at least about 3-to-1 to 6-to-1. The cream base and liquid activator each have a density of about 1, and thus the cream base to liquid activator volume ratio also is generally about 3-to-1 to 6-to-1.

The activator to be added to the cream base contains a significant amount of water in relation to the total amount of water present in the two components, and the cream base is usually formulated to be viscous so that when the additional water from the liquid activator is added to the cream base, the resulting admixed relaxer will have a manageable consistency and will not become runny. The viscous cream base, in itself, however, is thick and difficult to stir. Moreover, the large difference in rheologies between the cream base and liquid activator makes it more difficult to uniformly mix the activator with the cream base. Mixing times for conventional two-component systems are typically

long, on the order of two to four minutes, depending on the age of the cream base.

A food coloring is sometimes added to the activator so as to provide the consumer with visual feedback on the degree of mixing of the liquid activator and the cream base. For example, if red food coloring is added to the liquid activator, when mixed with a white cream base, the consumer will be directed to achieve a uniform pink color.

The two-component systems currently in use present a number of problems for the consumer. The existing two-component systems are most accurate when the user mixes all of the cream base supplied in the container with all of the concentrated liquid activator supplied in the bottle. Because these components are pre-measured to close tolerances by the manufacturer, the thorough mixing of full measures by the consumer results in relaxers whose chemical strengths are those designed by the manufacturer for different hair textures and types. Even when full measures are used, however, any accidental spillage (typically by splashing during mixing) of even a small amount of the concentrated liquid activator will yield a mixed relaxer having a lower strength than intended.

Another problem arises when the consumer uses only a portion of the relaxer kit at one time while saving the remaining product for later use. After having a relaxer treatment and as new hair grows, the consumer must relax the new growth to maintain the hairstyle. Because these touch-ups seldom require the entire amount of relaxer in a kit, the consumer typically will measure out and mix half of the contents of both the relaxer base cream container and the activator bottle. Then, the consumer saves the remaining unmixed components and uses them later for a second touch-up.

In preparing a relaxer using less than the full contents of a kit, the consumer generally relies on estimates which the consumer makes of half measures, from pre-drawn lines on the cream base container and the liquid activator bottle. For example, the consumer must first measure out one half the contents of the cream base. In some relaxers this is accomplished by measuring out a predetermined number of level scoops. Of course, if the consumer does not use the scoop and attempts to estimate one half the contents of the cream base, there is a large likelihood of error. Even if the consumer is careful in measuring out the indicated number of scoops, the

scoops may not be level, and the scoop may not be entirely emptied before refilling successive scoops.

Once having measured the one-half amount of cream base, the consumer must then measure out one half of the liquid activator containing guanidine carbonate. Such commercially available liquid activators typically contain a "half line" on the side of the bottle so that the consumer will know what is the half contents amount. Again, consumer errors in measuring amounts of activator are also common and difficult to avoid. Moreover, the half line is not always accurately placed on the bottle by the manufacturer, and the quantity of activator actually filled into the bottle may be over or under the intended amount due to mal-adjustment or malfunction of the high-speed filling equipment.

The problem arises that unless the consumer is extremely diligent in measuring out the one-half amounts of base and activator, the final guanidine hydroxide concentration in the mixture will greatly vary depending on whether the measured amounts are over or under the one-half target amount. It has been determined that consumer errors from measuring the base and/or activator can easily result in fluctuations of $\pm 10\%$ in the volume of cream base and $\pm 10\%$ in the volume of activator employed, resulting

in fluctuations in guanidine hydroxide concentrations of ± 10 percent or more, when the target amounts are half of the container contents. Measuring errors can be much greater when the estimated targets are other than half contents or when spillage occurs. These measuring errors of $\pm 10\%$ or more can lead to wide variations in strength which can yield poor results such as ineffective relaxing (e.g., the hair does not relax as much as it should) and premature reversion, when the concentration of guanidine hydroxide is too low, or excessive side effects such as scalp irritation, hair damage and hair breakage to the consumer, when the concentration of guanidine hydroxide is too high.

Typically, the cream base component of the two-component relaxer contains an excess of calcium hydroxide so that the reaction of the guanidine carbonate is driven to completion. It is therefore the amount of guanidine carbonate added to the cream base and the total volume of the final mixture that determines the concentration of the guanidine hydroxide in the product when it is applied to the hair. Measuring errors of components on the part of the consumer can greatly alter the concentration of guanidine hydroxide in the admixed product resulting

in insufficient relaxation or damage from overprocessing.

In addition to these errors in measuring the amount of liquid activator and cream base, errors are also introduced when the two components are mixed together. In order to obtain a uniform concentration of guanidine hydroxide in all portions of the relaxing mixture, the consumer must thoroughly mix the measured amount of activator and cream base.

It has been observed that consumer errors in the mixing step are also common and difficult to avoid. For example, if the consumer mixes the activator with only the top portion of the cream base, without diligently mixing the contents at the bottom of the cream base container, the concentration of guanidine hydroxide at the top portion of the container will be significantly higher than at the bottom portion of the container. The concentration of guanidine hydroxide at the top portion can vary significantly from that designed by the manufacturer, and can be, for example, as much as two times greater than that designed by the manufacturer. The insufficient mixing by the consumer creates a concentration gradient in the admixed relaxer. Thus, the first half of the relaxer placed on the hair will be much stronger than the bottom half of the

relaxer placed on the hair. This results in uneven relaxing and possibly damage to the hair.

U.S. Patent No. 5,068,101 discloses a two-component system in which one component is an aqueous solution of about 25 to about 30 weight percent
5 guanidine carbonate which serves as the liquid activator, and the other component contains about 4 weight% to about 7 weight% calcium hydroxide emulsified in a cosmetic cream base. About 3.5 to about 6
10 parts by weight of the cosmetic cream base are mixed with one part by weight of the activator to provide the guanidine hydroxide hair relaxer. U.S. Patent No. 5,077,042 discloses a conditioning activator which is substantially a liquid comprising a rela-
15 tively strong organic base and a highly alkaline, no-scalp-barrier- necessary, no-lye cosmetic cream containing calcium hydroxide.

These patents disclose the possibility that guanidine carbonate may be included in the emulsion
20 of the cream base and calcium hydroxide may be added in the form of an aqueous suspension just before use. U.S. Patent No. 5,077,042 describes this approach as being impractical, and states that the amount of calcium hydroxide required for the reac-
25 tion would be difficult to suspend in liquid form and would likely result in a non-uniform reaction

mixture which would not relax the hair properly. These patents do not disclose any example where the guanidine carbonate is in a cream base and the calcium hydroxide is in an aqueous suspension. These patents do not discuss the problem of consumer measuring and mixing errors.

As a result, these patents do not provide the consumer with a product which does not have the problems of variation in guanidine hydroxide concentration when mixed by the consumer.

Other problems associated with liquid activators include the possibility of spilling and splashing, which can affect the amount of activator that is added to the cream base. Spillage can cause stains, particularly if food coloring is present in the liquid activator.

Another problem in the art is the potential toxicity of the guanidine carbonate solution used to activate the relaxer cream. The relative toxicity of guanidine carbonate is well established in the literature. Currently, the guanidine carbonate solution used in a two-component relaxer is packaged in a child-resistant bottle to prevent accidental ingestion. However, given the volume of the solution (from 50 ml to 80 ml), it is possible that the entire contents of a bottle could be swallowed.

Therefore, the potential for accidental ingestion and poisoning based on the small amount of liquid guanidine carbonate remains a problem.

Food coloring has been added to the activator
5 in order to indicate the degree of mixing between the cream base and the activator. However, such a conventional type of indicator does not indicate that the guanidine carbonate has reacted with the calcium hydroxide. It merely shows the extent that
10 the liquid activator has been physically mixed with the cream base.

Another problem in the art is that ammonia is formed over time in the guanidine carbonate aqueous liquid activator component due to the hydrolysis of
15 the guanidine carbonate. The formation of ammonia in the guanidine carbonate aqueous liquid activator component causes an undesirable odor. Thus, the liquid activator itself is inherently unstable and has a relatively limited shelf-life.

20 A still further problem in the art is that relaxers are often irritating to the scalp of the user. When a consumer is also color treating the hair, which is also irritating to the scalp of the consumer, the consumer must wait for several weeks
25 after the relaxer treatment to color the hair (and vice versa) in order to avoid further irritation.

SUMMARY OF THE INVENTION

s Accordingly, an object of the present invention is to provide a two-component hair-relaxer system containing a cream base and activator which can
5 provide a concentration of alkaline hydroxide in the resultant admixture that does not vary significantly, even when errors are introduced by the consumer during the step of measuring amounts of cream base and amounts of activator.

10 A further object of the present invention is to provide a two-component hair relaxer system which can provide a more uniform concentration of alkaline hydroxide in the resultant admixture that does not vary significantly, even when errors are introduced
15 by the consumer during the step of physically mixing the cream base with the activator.

It is a further object of the present invention to provide a relaxer composition in which a potential toxic guanidine carbonate solution is present
20 in a form which reduces the likelihood of accidental poisoning by ingestion.

Another object of the present invention is to provide two components for a relaxer composition which are easier for the user to mix together.

25 A still further object of the present invention is to provide a water-containing composition which

contains a compound which is susceptible to hydrolysis and formation of ammonia in which the rate of formation of unwanted ammonia is reduced.

5 Another object of the present invention is to provide a relaxer that is not as irritating to the scalp and skin.

Yet another object of the present invention is to provide a relaxer composition which contains an indicator that provides the user with visual feedback that the cream base and the activator have
10 successfully reacted to form an efficacious relaxer.

To achieve the foregoing objects and in accordance with its purpose, there is provided, in a first aspect of the present invention, a two-component hair relaxer system for producing an alkaline hydroxide relaxer for relaxing hair comprising (a) a
15 first component comprising a water-containing cream base containing a water-soluble salt of a relatively strong base with an anion capable of being precipitated by an alkaline earth metal ion under highly
20 alkaline conditions, and (b) a second, separate component, which is substantially free of water, comprising an activator containing an alkaline material having an alkaline earth metal ion which forms
25 a precipitate with the anion when the first component and second component are mixed.

In a second aspect of the present invention, there is provided a water-containing composition which contains a water-soluble salt of a relatively strong nitrogen containing organic base with a carbonate anion and which salt is susceptible to alkaline hydrolysis and formation of ammonia, wherein the water-containing composition contains a carbonate/bicarbonate buffer to retard the decomposition and formation of ammonia.

In a preferred embodiment of the second aspect of the present invention, a pH indicator is present in the water-containing composition which contains a carbonate/bicarbonate buffer to provide the user with a reliable indication of the thoroughness of mixing by changing color when the water-soluble salt of the relatively strong nitrogen containing organic base with a carbonate anion has reacted with an alkaline material having an alkaline earth metal ion which forms a precipitate with the carbonate anion.

It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory, but are not restrictive of the invention.

DETAILED DESCRIPTION OF THE INVENTION

In the first aspect of the present invention, there is provided a hair relaxer system of the present invention which comprises (a) a first component
5 (the cream base) comprising a water-containing cream base containing a water-soluble salt of a relatively strong base with an anion capable of being precipitated by an alkaline earth metal ion under highly alkaline conditions, and (b) a second component (the
10 activator) which is substantially free of water, and containing an alkaline material having an alkaline earth metal ion which forms a precipitate with the anion when the first component and second component are mixed.

15 The cream base of the present invention preferably is an oil-in-water emulsion and contains a water-soluble salt of a relatively strong base with an anion capable of being precipitated by an alkaline earth metal ion under highly alkaline conditions.
20 The water-soluble salt is present in the aqueous phase of the oil-in-water emulsion.

The relatively strong base which forms the water-soluble salt is a base which provides a pH of between 12 and 14 when in an aqueous medium. The
25 relatively strong base generally is an organic nitrogen containing base, and preferably is guanidine.

Other organic bases which may be used in place of guanidine include N-methyl guanidine, dimethylamino-guanidine, acetamidine, dimethylaminoamidine, amino-amidine and acetamide.

5 The anion capable of being precipitated by an alkaline earth metal under highly alkaline conditions preferably is the carbonate ion. Thus, the preferred water-soluble salt is guanidine carbonate. Salts other than the carbonate salt, such as a sul-
10 fate, sulfite, phosphate, fluoride, oxalate, tartrate, laurate or alginate salt can be used.

 The oil phase generally contains anhydrous, lipophilic ingredients, which include an oleaginous material and an emulsifier.

15 Any suitable oleaginous material can comprise the oil phase for the cream base emulsion. Suitable oleaginous materials include petrolatum, mineral oil and mineral jellies, but also can include vegetable and animal derived oils and fats, and like unctuous
20 emulsifiable materials. Preferred oleaginous materials are mineral oil and petrolatum or a mixture of the two. Particularly preferred is a petrolatum-mineral oil mixture, where the petrolatum and mineral oil are present in approximately equal weight
25 amounts. At least 50 weight percent, preferably about 55 to about 75 weight percent of the oil phase

is comprised of the oleaginous material such as the mineral oil-petrolatum mixture.

The oil phase may further comprise emulsifiers, present in an amount of about 5 to about 18 weight percent of the cream base. Suitable emulsifiers include nonionic emulsifiers, anionic emulsifiers, cationic emulsifiers, and amphoteric emulsifiers.

Suitable nonionic emulsifying agents include fatty alcohols, polyoxyethylene derivatives of fatty acid esters of sorbitol and sorbitol anhydride; polyethylene glycol esters of fatty acids, polyoxyethylene ethers of fatty alcohols, polyethylene oxide-polypropylene oxide condensates and polyoxyethylene lanolin ethers, and the like.

Examples of preferred non-ionic emulsifiers include a mixture of fatty alcohols having about 12 to about 24 carbon atoms, preferably 14 to about 22 carbon atoms in the fatty carbon chain. Specific examples include cetyl alcohol, stearyl alcohol, Steareth-10, Steareth-20, PEG-5 Soya sterol, and PEG-10 Soya sterol. Other suitable emulsifiers which may be added to the oil phase include cetareth-20 (a non-ionic emulsifier comprised of a polyethylene glycol ether of cetearyl alcohol, which is a mixture of cetyl and stearyl alcohol). Cosmo-wax J is a preferred non-ionic emulsifier, sold by

Croda, Inc., New York, New York. Cosmowax J is comprised of 80% cetearyl alcohol and 20% ceteareth-20. Cetearyl alcohol is a mixture of fatty alcohols containing predominantly cetyl alcohol and stearyl alcohol. Ceteareth-20 is a non-ionic emulsifier comprised of a polyethylene glycol ether of cetearyl alcohol. The non-ionic emulsifier generally is present in an amount of about 6 to 14 weight percent, preferably 8 to about 12 weight percent of the cream base.

Suitable anionic emulsifying agents include polyoxyethylene oleyl phosphates, sodium lauryl sulfate, and the stearic acid anion and the like. Polyoxyethylene (3) oleyl ether phosphate is particularly preferred.

The oil phase comprises about 15 to about 55 weight percent, preferably about 25 to about 45 weight percent of the cream base. The oil phase of the cream base is emulsified with the aqueous phase of the cream base which is described as follows.

The aqueous phase of the cream base comprises water and the water-soluble salt of a relatively strong base (e.g., guanidine carbonate) which can be added in suitable amounts.

The concentration of water-soluble salt (e.g., guanidine carbonate) in the cream base is generally from 1 to 20% by weight, preferably from 2 to 15% by weight. More preferred concentrations of the water-soluble salt (e.g., guanidine carbonate) are from 1 to 10% by weight and most preferred is 6 to 8% by weight of the cream base.

The water may be present in the aqueous phase in amounts of 80 to 90 weight %, and in the cream base in amounts of 40 to 60 weight %. Water preferably comprises at least 50 weight percent of the cream base.

One or more co-emulsifiers may be added to the aqueous phase of the cream base to improve the texture of the cream base and control viscosity. A particularly preferred co-emulsifier is PPG-12 PEG-65-Lanolin oil which is a polyoxypropylene, polyoxyethylene derivative of lanolin oil, such as sold under the tradename Fluilan AWS by Croda, Inc., New York, New York. Lanolin derivatives may be added in an amount of about 1 to about 3 weight percent of the cream base.

A conditioner may be added to the cream base as desired. Suitable conditioning agents include non-polymeric quaternary nitrogen containing compounds such as those taught in U.S. Patent No. 5,077,042.

Particularly preferred non-polymeric quaternary nitrogen containing conditioners include Finquat CT (Quaternium 75 sold by Fintex, Inc., Elmwood Park, New Jersey) and Arquad 2HT-75 (Quaternium 18 manufactured by Akzo Chemical, Inc., Chicago, Illinois). Amounts of conditioner are preferably about 0.05 to about 5 weight percent, more preferably about 0.1 to about 4 weight percent of the cream base. Although polymeric quaternary nitrogen containing conditioners can also be used, they are not preferred for use in a guanidine carbonate-containing cream base of the present invention since they usually result in an unstable emulsion which separates upon aging and which must be remixed before use.

A surfactant may be added to the cream base as needed to enhance the rinsing of the relaxer from hair. The surfactant can comprise anionic or amphoteric compounds such as dioctyl sodium sulfosuccinate, lauroyl sarcosine, and cocoamphopropyl sulfonate.

Preferred surfactants for use with guanidine carbonate include Crodafos SG, Miranol C2M-SF, Miranol C2M, Sandopan DTC, and Duponol XL. Crodafos SG is PPG-5 ceteth-10 phosphate sold by Croda, Inc., New York, New York. Miranol C2M-SF is disodium cocoamphodipropionate sold by Miranol, Inc., Dayton,

New Jersey. Miranol C2M is disodium cocoamphodi-
ace-tate, also sold by Miranol. Sandopan DTC is
sodium trideceth-7 carboxylate sold by Sandoz Chemi-
cal Corporation, Charlotte, North Carolina. Duponol
5 XL is a surfactant containing DEA-lauryl sulfate,
sodium lauraminopropionate and DEA-lauraminoproprio-
nate, sold by Dupont Company, Wilmington, Delaware.

The surfactant may be present in the cream base
in an amount of from 0.5% to 4%, preferably 1% to
10 3%, more preferably 1% to 2%, by weight of the cream
base.

The cream base can also include cosmetic adju-
vants, such as auxiliary emollients, auxiliary
thickening agents, perfumes, preservatives, and
15 product colorants.

The total amount of non-water components in the
cream base preferably makes up no more than about
50% by weight of the total weight of the cream base.

In preparing a typical cream base component
20 according to the present invention, guanidine car-
bonate is present in the cream base emulsion prefer-
ably in amounts of between 6 to 8 percent by weight.
The guanidine carbonate may be added to the cream
base as a dry powder. In preparing the cream base
25 composition, the oil phase components and the water
phase components without guanidine carbonate are

heated in separate vessels to between 70°C and 80°C. Then, the oil phase is added to the aqueous phase and homogenized at high speed for about 15 minutes to about 30 minutes. When the mixture has cooled to
5 a temperature of about 45°C, the guanidine carbonate is added and stirring is continued while a conditioner, if desired, is added. Then the surfactant is added. Alternatively, the guanidine carbonate can be added to the water phase before the water
10 phase is mixed with the oil phase.

The second component of the two-component relaxer system of the present invention is the activator, which is substantially free of water, and which contains an alkaline material having an alkaline
15 earth metal ion which precipitates with the anion of the water-soluble salt present in the cream base. The alkaline material in the activator can be an alkaline earth metal hydroxide such as calcium hydroxide, barium hydroxide and strontium hydroxide,
20 or an alkaline earth metal oxide, such as calcium oxide or barium oxide, which forms the corresponding alkaline earth metal hydroxide when placed in an aqueous medium, or a mixture of the alkaline earth metal hydroxide and alkaline earth metal oxide.
25 Calcium hydroxide or a mixture of calcium hydroxide

and calcium oxide is the preferred alkaline material in the activator of the present invention.

When the preferred calcium hydroxide activator is admixed with the preferred cream base containing
5 guanidine carbonate prior to use, insoluble calcium carbonate and soluble guanidine hydroxide are formed.

The activator of the present invention, which is substantially free from water, is in the form of
10 a powder or a lotion.

As used herein, the term "substantially free of water" means that small amounts of water of less than about 20 weight percent, preferably less than about 10 weight percent, can be present in the acti-
15 vator, as long as the water does not interfere with the ability of the activator to flow freely when in powder form or pour easily when in lotion form. Generally, it is preferred that no water be present in the activator so that the activator is anhydrous,
20 but small amounts of from about 1 to about 20 weight percent can be tolerated.

When the activator is in the form of a powder, the powder can contain up to about 20 weight percent by water and still be a flowable powder. Greater
25 amounts of water in the powder result in a thick, non-free flowing non-pourable mass up to about 50%

water. At about 50% water and above, the alkaline material would be present as an aqueous suspension. When the activator is in the form of a lotion, the lotion can contain up to about 20% weight percent water without substantially affecting the properties of the lotion, but in amounts of more than about 20%, the water would change the viscosity of the lotion and the mixture would become an unpourable, unworkable semi-solid mass.

When in the form of a lotion, the activator is a viscous, pourable suspension which contains the alkaline material, such as alkaline earth metal hydroxide, for example, calcium hydroxide, generally in an amount of from 20 to 60 weight percent, preferably 30 to 55, most preferably from 40 to 50 weight percent. If the amount of alkaline material such as alkaline earth metal hydroxide in the lotion is more than 50 weight percent, the lotion does not pour well. The alkaline material such as alkaline earth metal hydroxide is present in the lotion in an amount sufficient to provide a molar excess of the alkaline material, such as alkaline earth metal hydroxide, with respect to the water-soluble salt of the cream base when mixed with the cream base. Generally, a molar excess of alkaline material (e.g., calcium hydroxide) of from about 5% to about

200% can be employed, more preferably 10 to 120%. It will be understood that the term "molar excess" as used herein refers to an amount of alkaline material which is in excess of the stoichiometric amount of alkaline material needed to react with the water-soluble salt.

The activator lotion contains a suitable water-miscible, hydrophilic liquid carrier to deliver the alkaline material (e.g., calcium hydroxide) present in the activator into the aqueous, continuous phase of the cream base emulsion, such as a guanidine carbonate containing cream base emulsion. Suitable carriers include polyhydroxy compounds (such as propylene glycol, glycerine, butylene glycol and hexylene glycol), and ethers including simple ethers and polyethers. A preferred carrier is propylene glycol. The propylene glycol also acts as a humectant in the hair to prevent dryness.

The amount of carrier in the lotion is preferably from 40 to 80% by weight, more preferably 50% to 60%.

Additionally, the activator lotion may contain a desiccant in order to remove any water that may be present in the activator and to maintain the activator lotion substantially anhydrous until the time of its use. Commercially obtained solid calcium hy-

droxide powder may sometimes contain small amounts of water, as shown by an off-white color, and the addition of a desiccant to the lotion, insures that the lotion will be substantially free of water.

5 Suitable desiccants include simple anhydrides, polymeric anhydrides, or molecular sieves. A preferred desiccant is calcium oxide because it becomes calcium hydroxide when mixed with water.

10 The desiccant is generally present in the lotion in amounts of from 0 to 20% by weight, preferably 1% to 15%, more preferably 3 to 10%. When using calcium oxide as a desiccant, the amount thereof is included in the determination of the amount of alkaline material present in the activator.

15

20 The activator lotion may also contain a coloring agent or opacifying agent. Preferred coloring agents are pigments and dyes. Suitable pigments include metal oxide whiteners. A preferred whitener is titanium dioxide. The whitener is preferably present in the lotion in an amount of from 0.5 to 5% by weight.

25 In addition, the lotion activator may contain a thickener. Suitable thickeners include organic polymers such as hydroxypropyl cellulose and hydroxyethylcellulose, anhydrous aluminum silicate

and hydrated magnesium aluminum silicate and colloidal clays. A preferred thickener is fumed silica. The thickener is preferably present in the lotion in amounts of from 1 to 6% by weight, more preferably, 2% to 5% by weight.

The activator lotion of the present invention is a phase-stable composition which when subjected to an accelerated aging test at 50°C for 30 days shows no phase separation. The term "phase-stable" as used herein refers to the physical stability of the lotion. The phase-stable lotions of the present invention do not visibly separate when subjected to the above accelerated aging test. The above time period represents a commercially useful lifetime in the field, and the lotion is stable in the field for a period of at least one year.

When in the form of a powder, the activator powder comprises a powder or crystalline form of the alkaline material, such as solid calcium hydroxide powder or calcium oxide powder. The alkaline material (e.g., calcium hydroxide) preferably has a particle size such that >95% passes through a 325 mesh sieve. Amounts of alkaline material (e.g., calcium hydroxide) in the powder are preferably from 20 to 100% by weight, still more preferably from 60% to 90% by weight. A preferred amount of alkaline mate-

rial (e.g., calcium hydroxide) in the powder is from a 5% to a 200% molar excess over the water-soluble salt (e.g., guanidine carbonate) in the relaxer cream base, more preferably a 10% to a 120% % molar excess.

A surfactant/wetting agent may be present in the activator powder in order to provide the powder in a non-dusty state (i.e., to promote its pourability) and to promote the dispersability of the powder in the cream base. The surfactant/wetting agent can be present in the powder in amounts of from 2 to 25% by weight, preferably 10 to 20% by weight. Suitable surfactants/wetting agents include nonoxynols, sorbitols, and alkyl pyrrolidones. A preferred surfactant for use in the activator when it is in the powdered form is Surfadone LP 100 (ISP, Inc., Wayne, New Jersey). The powder can be prepared by mixing calcium hydroxide powder with the surfactant by methods well known in the art, such as ribbon blending.

Other inactive ingredients can be present in the powder, such as calcium carbonate which serves to prevent compaction of the calcium hydroxide powder particles.

In a preferred embodiment of the present invention, the activator is in the form of a lotion that

is substantially free of water. This form is preferred from the standpoint of easier mixing because the activator lotion has a closer rheology to the cream base.

5 The ratio of alkaline material containing activator (such as alkaline earth metal hydroxide containing activator) to cream base is generally from one part by weight activator to about 6 to 30 parts by weight cream base, most preferably 10 to 15 parts
10 by weight cream base. The density of the cream base is generally about 1 gm/ml. The density of the activator is generally from about 1.4 gm/ml (for a lotion activator) up to about 2.34 gm/ml for a powder activator. The volume ratio of activator to
15 cream base is generally from about one part by volume activator to about 8 to 50 parts by volume cream base, such as 10 to 25 parts by volume cream base.

 The use of an activator lotion or powder that is substantially free of water is designed to deliver
20 to the relaxer composition the maximum amount of the alkaline material having an alkaline earth metal ion (e.g., calcium hydroxide or calcium oxide) possible with the least amount of added volume. Because the activator to be added to the cream base is
25 substantially free of water, the total amount of water in the final mixture will be that delivered

from the cream base, and will remain relatively unchanged regardless of the ratio of activator to cream base which the consumer uses. Moreover, because the activator is in the form of a powder or a lotion which contains a high concentration of the alkaline material, the volume of activator necessary to supply the desired amount of alkaline material is relatively small, especially as compared to the volume of cream base. Therefore, the total volume of the admixture of cream base and activator will not be substantially affected by errors in volume in the amount of the activator and in the amount of the cream base.

Because the cream base will contain a fixed concentration of guanidine carbonate, the concentration of guanidine hydroxide in the product applied to the hair will remain relatively constant. Excess calcium hydroxide added to the base would remain insoluble and therefore, not have an appreciable effect on the concentration of the guanidine carbonate in the admixture.

In the conventional prior art method, a guanidine carbonate liquid activator is added to a calcium hydroxide cream base. Because the concentration of guanidine carbonate in such a relaxer is a function of the volume and concentration of the activa-

tor and the volume of the base, and because the activator is present in a volume amount of usually 1/3 to 1/6 the volume of the cream base, measuring errors in the amount of activator greatly affect the concentration of guanidine carbonate in the relaxer. The concentration of guanidine carbonate (Cr) (gm/ml) in the relaxer when the guanidine carbonate is supplied from a liquid activator as in the prior art can be defined by the following equation (Eq. 1):

$$Cr = \frac{Va \times Ca}{Va + Vb} \quad (\text{Eq. 1})$$

where Va = volume of activator

Ca = concentration of guanidine carbonate in the activator (gm/ml)

Vb = volume of base

It can be seen that measuring errors can affect the volume of activator and volume of base. Because the activator is in the form of a concentrated aqueous solution in the prior art and is present in a volume amount of usually about 1/3 to about 1/6 the volume of the cream base, the amount of activator used has a large effect on the total volume of the admixture and thus the concentration of guanidine carbonate (Cr) in the relaxer.

In the present invention, however, the guanidine carbonate is present in the cream base at a

fixed concentration and the calcium hydroxide in the activator is substantially free of water. The concentration of guanidine carbonate (Cr) (gm/ml) in the relaxer when the guanidine carbonate is supplied in the cream base according to the present invention can be defined by the following equation (Eq. 2):

$$Cr = \frac{Vb \times Cb}{Vb + Va} \quad (\text{Eq. 2})$$

where Vb = volume of cream base
 Cb = concentration of guanidine carbonate in cream base (gm/ml)
 Va = volume of activator

Because the volume amount of activator is usually 1/50 to 1/8 the volume amount of cream base, when the activator is added to the cream base, the effect of the activator on the total volume is very small. As a result, errors in the amount of activator and cream base do not result in significant errors in the final concentration of guanidine carbonate, when only a part of the cream base is measured out by the user.

In the present invention, if the consumer adds the activator properly but fails to mix the relaxer thoroughly from top to bottom, the concentration of the alkaline guanidine hydroxide in the top portion will not exceed the intended concentration. Because hair damage and scalp irritation can be serious

consequences of using a too-strong relaxer preparation, the present invention has very significantly improved the safety of these mix-type relaxers.

5 In the two-component system of the present invention, the rheology of the two components is more closely matched than conventional two-component mix systems, and the mixing shear is improved. Further, the present system does not use an aqueous free-flowing liquid activator, and thereby reduces
10 problems such as splashing and spillage of the activator and viscosity differences between activator and cream base. In addition, because the activator is substantially free of water, more water may be present in the cream base which results in a softer
15 consistency for the cream base. This softer consistency makes it easier for the consumer to mix the activator into the base.

The provision of guanidine carbonate in a cream base emulsion reduces the likelihood of accidental
20 ingestion of a toxic amount of guanidine carbonate because the guanidine carbonate is present in the cream base in a lower concentration than in prior art liquid activators, and the volume of cream base which contains the guanidine carbonate is significantly higher than the volume of prior art liquid
25 activator compositions employed in a two-component

system. Thus, if one were to ingest the same volume amount of cream base as liquid activator, then one would be receiving a lower dosage of the guanidine carbonate. Moreover, the volume of cream base which
5 would be present in a two-component relaxer system of the present invention is so large that it is highly unlikely that a child would accidentally ingest an amount which is sufficient to be toxic. Further, the prior art liquid activators are in the
10 form of a solution which is easier to ingest than a cream base.

In a second aspect of the present invention, there is provided a water-containing composition which contains a water-soluble salt of a relatively
15 strong organic nitrogen containing base with a carbonate anion and which is susceptible to alkaline hydrolysis and formation of ammonia, wherein the water-containing composition contains a carbonate/bicarbonate buffer to retard decomposition of
20 the water-soluble salt and formation of ammonia. The carbonate/bicarbonate buffer is present in the composition in an amount which provides the composition with a pH of from about 9.5 to 10.5.

The water-containing composition which is provided with the carbonate/bicarbonate buffer can be
25 the cream base component of the first aspect of the

present invention and wherein the water-soluble salt of the first aspect of the present invention is a water-soluble salt of a relatively strong organic nitrogen containing base with a carbonate anion and which is susceptible to alkaline hydrolysis, or can be a liquid activator such as used in the prior art wherein the liquid activator comprises a water solution of a water-soluble salt of a relatively strong organic base with a carbonate anion (such as guanidine carbonate), and which is susceptible to alkaline hydrolysis and which is present in an amount of about 20 to 30% by weight. Such a liquid activator can be a conventional liquid activator such as described in U.S. Patent No. 4,304,244 and can contain conventional additives such as polyhydroxy compounds, thickening agents, metal-ion chelating agents, preservatives and perfume and can be used with conventional cream bases containing an alkaline material which reacts with the water-soluble salt, such as described in U.S. Patent Nos. 4,304,244, 5,068,101 and 5,077,042.

For purposes of illustration only, the following description of the second aspect of the present invention will be based on the use of guanidine carbonate as the water-soluble salt, but is equally applicable to carbonate salts of any of the other

organic nitrogen containing bases which have previously been described.

The pH of a cream base of the first aspect of the present invention containing guanidine carbonate is about 11.5 to 12. The guanidine carbonate in this cream base slowly decomposes over time at ambient temperature to liberate ammonia due to hydrolysis with the water present in the cream base. A strong objectionable ammonia odor is formed when such a cream base is packaged. Similarly, the guanidine carbonate liquid activators of the prior art have a pH of about 11.2 to 12.0 and also suffer from decomposition and formation of ammonia odor due to hydrolysis with water.

It has now been found in accordance with the second aspect of the present invention that the decomposition and formation of ammonia at ambient and elevated temperature can be retarded by lowering the pH of the water-containing component (which for ease of explanation is referred to hereafter as the guanidine carbonate component) which contains the water-soluble carbonate salt of a relatively strong organic nitrogen containing base and which is susceptible to alkaline hydrolysis and formation of ammonia to about 10.5 or less, preferably to about 9.5 to 10.4.

In order to lower the pH of the guanidine carbonate component, a carbonate/bicarbonate buffer is created in the guanidine carbonate component by the addition of a suitable proton donor, such as by the addition of guanidine bicarbonate. When 5 to 10 mole percent of the guanidine carbonate is present as guanidine bicarbonate, the pH remains below a value of 10.4.

Guanidine bicarbonate may be added directly to or substituted for the guanidine carbonate in the guanidine carbonate component in an amount of from 0.4 to 5 percent by weight of the guanidine carbonate component or may be formed in situ by adding other relaxer components in their acid form.

Another method for lowering the pH of the guanidine carbonate component (either cream base or liquid activator) is to add a suitable acid to the guanidine carbonate component. A suitable acid is one that lowers the pH of the guanidine carbonate component without interfering with the relaxing process. An indication that the acid has interfered with the relaxing process is when excessive reversion occurs. An acceptable amount of reversion for a commercial product generally is about 15% or less, preferably less than 10%. Reversion is measured by the procedure in Example 4. For best results, the

acid should not cause a degree of reversion of more than 10%.

The acid which is used may be an organic acid or an inorganic acid. Examples of suitable organic acids include aliphatic acids, alicyclic acids, aromatic acids and heterocyclic acids. These acids may be substituted. Examples of suitable substituents include an amino group and a hydroxy group. Suitable acids include monocarboxylic acids and dicarboxylic acids. Suitable dicarboxylic acids include substituted and unsubstituted aliphatic dicarboxylic acids, such as succinic acid, maleic acid, oxalic acid, and glutamic acid. Suitable monocarboxylic acids include substituted and unsubstituted aliphatic monocarboxylic acids, such as lactic acid. Suitable inorganic acids include sulfuric acid and phosphoric acid.

Preferred acids include succinic acid, maleic acid and glutamic acid.

In general, the amount of acid in the water-containing composition of the second aspect of the present invention is from about 5 to 20 mol%, preferably 5 to 10 mol%, of the number of mols of water-soluble carbonate salt (e.g., guanidine carbonate) in the water-containing composition.

In the preferred method for preparing the cream base containing the acid, the guanidine carbonate is first dissolved in the aqueous phase of the cream base, and then the acid component is added to form the carbonate/bicarbonate buffer.

Ammonia is formed at ambient or elevated temperatures due to the presence of hydroxide ions when the pH of the guanidine carbonate component is around 11.2 to 12, but it surprisingly has been found that by lowering the pH in the guanidine carbonate component of a two-component relaxer system to about 10.5 or less, the rate of formation of ammonia at ambient and elevated temperature is greatly reduced.

Surprisingly, in accordance with the second aspect of the present invention, it has also been discovered that lowering the pH of the guanidine carbonate component to retard ammonia formation significantly reduces skin and scalp irritation when the admixed relaxer composition is applied. The present invention has discovered that free ammonia is a significant contributor to skin and scalp irritation in guanidine-based relaxer systems. The fact that ammonia is a significant contributor to scalp irritation was not previously known in the art, and it was generally believed that the side-effects of

stinging and burning sensations were principally caused by the guanidine hydroxide.

Further, it has been discovered that ammonia causes stinging and burning sensations even when present in the shampoo component that is applied to neutralize the relaxer after it has been rinsed from the hair. Accordingly, in a preferred embodiment of the present invention, a neutralizing shampoo is employed which does not contain any component which is subject to alkaline hydrolysis and formation of ammonia.

In a further embodiment of the second aspect of the present invention, a pH indicator is present in the water-containing composition of the second aspect of the present invention, which contains a carbonate/bicarbonate buffer in order to provide an indication to the user of the thoroughness of the mixing of the cream base and the activator. The color change of the pH indicator signals that a pH increase has occurred when the guanidine carbonate reacts with the calcium hydroxide. Thus, a uniform color change not only indicates thorough mixing but actually signals the presence of guanidine hydroxide so that the consumer knows that the ingredients of the product are active and that a working relaxer is present. The pH indicator can be present either in

the cream base of the present invention which is provided with a pH of about 9.5 to 10.5, or in the guanidine carbonate liquid activator of the prior art.

5 A pH indicator would not work with a conventional two-component system of the prior art because there is an insufficient pH gradient between the pH of the conventional calcium hydroxide cream base (generally about 12.2 to 12.5) and the mixing pH of
10 the admixed relaxer (generally about 13.5 to 13.7). High-pH indicators typically require a pH gradient of at least two.

 In the second aspect of the present invention the guanidine carbonate component is provided with a
15 pH of about 9.5 to 10.5, and a pH of the admixed relaxer is about 13.5 to 13.7, so that there is a sufficient pH difference which permits the use of known pH indicators. Therefore, standard pH indicators known to those skilled in the art can be used
20 to provide the user with feedback as to the degree of mixing. In particular, a uniform color change will indicate thorough mixing. Suitable pH indicators include Tropaeolin "O" (also known as, Acid Orange 6), Thiazole Yellow "G", Alizarine Yellow R, and Metachrome Yellow. Other pH indicators that are
25 suitable for use in the present invention include

Eosin I, Poirriers Blue, Mordant Blue #1, Mordant Red #3, and External D&C Violet #2.

5 The pH indicator is typically added in an amount of from 0.0005% to 0.5% by weight, preferably 0.0008 to 0.01, most preferably 0.0015 to 0.005% by weight of the water-containing composition.

10 Preferably a kit for a conditioning hair relaxer system embodying the principles of this invention comprises at least two packages. For example, a first package can include the cream base component (e.g., the guanidine carbonate containing cream base) as described above. A second package can include the activator component (e.g., the calcium hydroxide containing activator). The contents of
15 the first and second packages are admixed to provide an active relaxer composition just prior to use. In order to further minimize measuring errors, the activator component can be packaged in two separate packages, with each package containing one half of
20 the total activator component. Then, when a consumer wishes to use the full contents of the kit, the consumer will empty the contents of both packages containing the activator composition into the contents of the first package. When the consumer wishes
25 to use the half content of the kit, the consumer will measure out half the content of cream base

package in a conventional manner and then empty one of the packages containing the activator into the measured half content of the cream base package. Since a single activator package contains exactly one half of the full amount of activator composition, no measuring error with respect to the activator is possible. The activator can be formulated to contain a 100% or greater molar excess of alkaline material, such as a 110% molar excess. If the activator component is then packaged in two separate packages, each package will contain the necessary number of moles to react with the full content of the cream base package. Thus, even if a consumer mistakenly uses a single package of activator with the full contents of the cream base package, there will be sufficient alkaline material to react with all of the water-soluble salt in the cream base.

The time of treatment of hair to be relaxed with the relaxer formulation of the present invention will normally be within the range of 5 to 45 minutes, starting from the first application of the relaxer composition to the hair. Generally, this treatment time will be at least 10 minutes, and there is normally no real upper limit on the time that the composition can remain on the hair. It is preferred to treat the hair for no more than about

30 minutes, preferably less than 25 minutes, and more preferably around 20 minutes.

After the above treatment time has elapsed, the relaxer composition should be removed from the hair in order to prevent damage to the treated hair. A major portion of the relaxer composition can be removed from the hair by thorough rinsing. It is preferred that the rinsing be followed by a neutralizing step, using any suitable agent that will neutralize alkali.

Further details of how to use a relaxer are disclosed in U.S. Patent No. 4,373,540, which patent is hereby incorporated by reference.

The two-component relaxer system of the present invention provides numerous beneficial properties, which include low or no chemical odor, no-mistake lotion or powder activator, much easier mixing, very minimal scalp irritation, if any, faster working formula, more effective straightening of resistant hair, no relaxer smell left in hair, and very minimal reversion. Because the two-component relaxer system of the present invention substantially eliminates and reduces the problems caused by user measuring/mixing errors, it is highly reliable in that it always gives the same predictable good results each time the two-component relaxer system is used.

In the two-component hair relaxer system of the present invention, the water-soluble salt of a relatively strong base with an anion capable of being precipitated by an alkaline earth metal ion is the strength-determining chemical, and is formulated in the relaxer cream base at precisely the concentrations required for different hair types and textures; whereas, in the prior art it was contained in the liquid activator. The activator of the present invention is a super concentrate so that a small amount of the activator is all that is required to react with the water-soluble organic salt in the base. Using more of the activator than is the minimum required to react with the water-soluble salt of the cream base does not substantially affect the relaxer's strength or the results on the hair and scalp. Because the activator contains an excess of the alkaline material, the chance that too little of the activator will be used is highly remote.

It is, of course, important that the consumer stir/mix the activator and the cream base thoroughly to bring about the desired reaction. The new relaxer base has been designed to ensure good mixing results. First, the new base is very light, smooth, and creamy, and it can be stirred with ease. Second, the relaxer base preferably contains a color

signal component that appears automatically as the base is stirred signalling that the water-soluble salt in the cream base has been reacted with the activator.

In the following examples, all amounts are by weight unless otherwise indicated.

EXAMPLE 1

A two-component hair relaxer system according to the present invention was formulated, with one component being in the form of a cream base and the other component being in the form of a lotion. The components had the following compositions.

Formulation of Relaxer Base Containing Guanidine Carbonate.

	<u>Oil Phase</u>	<u>Weight Percent</u>
	Cosmowax J	12.00
	Petrolatum	10.00
	Light Mineral Oil	10.00
	PEG-5 Soya Sterol	1.00
	(sold under the name Generol 122E-5 by Henkel Corp., Hoboken, N.J.)	
	<u>Water Phase</u>	
	Deionized Water	54.7985
	PPG-12 PEG-65-Lanolin Oil (sold under the name Fluilan AWS by Croda, Inc.)	3.00
	Guanidine Carbonate	7.40
	Succinic Acid	0.30
	Thiazole Yellow "G" (sold by Pylam Products Co., Inc., Garden City, New York)	0.0015
	PPG-5-Ceteth-10 Phosphate (sold under the name Crodafos SG by Croda Inc.)	1.50
		<hr/> 100.00

The pH of the relaxer base is 10.1.

Formulation of Activator Containing Calcium Hydroxide

	<u>Weight Percent</u>
5	
Propylene Glycol	51.0
Silica (Cabosil M5 sold by Cabot Corp., Tuscola, IL)	4.0
10 Calcium Hydroxide	37.0
Calcium Oxide	6.0
10 Titanium Dioxide	2.0

The density of the activator lotion is 1.429 grams per milliliter.

15 To prepare the cream base, the components of the oil phase are placed together in a heatable vessel, and are heated to between 75 and 80°C.

In a separate heatable vessel, the above components of the water phase are added, and homogenized.

20 The Fluilan AWS should be heated to 60 to 65°C before it is added to the heatable vessel. The guanidine carbonate is thoroughly dissolved in the water phase, and then succinic acid is added to the water phase.

25 The vessel containing the water phase is then heated quickly to 70°C, and the heated oil phase is then added slowly to the water phase with moderate homogenization. The resulting emulsion is homogenized for 15 minutes more with moderate agitation.

30 The homogenized emulsion is then transferred to a cooling vessel where it is with stirring cooled to 50°C. At 50°C, the Crodafos SG can be added, and

mixing and cooling continue. The Thiazole Yellow G (a color indicator) can be added when the temperature reaches 45°C, and mixing is continued.

5 To prepare the activator, propylene glycol is placed in a homomixer and then moderately agitated. To the propylene glycol is slowly added the Cabosil M5 and mixed at high shear for approximately 30 minutes or longer until fully dispersed. Then, the calcium hydroxide and calcium oxide are added to the
10 batch and mixed for approximately 25 minutes or until uniform. Next, the titanium dioxide is added and mixing is continued for 35 minutes to obtain the activator lotion.

15 To form the hair relaxer, the cream emulsion and the activator paste are admixed under ambient conditions in a weight ratio of 12.5:1 of cream emulsion:activator lotion. A spatula can be used to form a well in the cream emulsion, and the lotion added to the cream. The spatula is used to mix the
20 cream base and activator lotion together thoroughly, scraping the sides and bottom of the mixing container, until the resulting relaxer composition is creamy and has a uniform color, free of streaks. This mixing takes about 2 minutes.

25 The hair of an individual to be treated is gently combed to remove tangles, and then the hair

is sectioned, and a generous amount of the relaxer formulation is applied to the hair on a section-by-section basis. The hair is combed gently after the relaxer is applied to ensure even distribution, with
5 care taken to avoid pulling or stretching the hair. When the entire head appears to be covered by the relaxer cream formulation, the hair is parted with a comb in different areas of the head and checked to ensure that the relaxer formulation has penetrated
10 to the hair root area. Thereafter, all of the hair is smoothed down, from front to back, using hands or the back of a plastic comb.

The timing of the length of hair relaxer application is started from the moment of first application of the relaxer formulation, and the hair is
15 treated for 15 - 20 minutes. For coarser textures than the medium textured individual tested, the maximum recommended time would be 20 - 25 minutes, whereas for individuals with finer hair, a treatment
20 time of 10 - 15 minutes might suffice. It has generally been noted that porous hair or hair that has been colored requires less time than untreated hair.

After the desired treatment time had elapsed, the hair is rinsed thoroughly with warm water and
25 the hair is then neutralized to approximately a

neutral pH by shampooing with a buffered neutralizing shampoo.

5 The neutralizing shampoo has a pH of 4 to 5, a buffer capacity of 0.1 to 0.2 milliequivalents of 0.10 N NaOH per gram of shampoo, and is substantially free of ammonia and ammonium ions.

10 The shampooing involves two lather and rinse cycles, and after the shampooing step the hair is optimally treated with a protein conditioner. After a final rinse the hair is towelled dry and set.

15 The hair treated in this example has a permanent relaxing or straightening effect, which lasts until new growth appears at the hair roots. The application of this relaxer composition to the individual's scalp, ears, and other parts of the body results in reduced complaints of skin irritation and burning sensations.

Example 2

20 A two-component hair relaxer system according to the present invention was formulated with the following composition for the cream base.

Formulation of Relaxer Base Containing Guanidine Carbonate

	Ingredient	Weight %
5		
	<u>Oil Phase</u>	
	Cosmowax J	10.50
10	Mineral oil	10.75
	Petrolatum	10.75
	PEG-5 Soya Sterol	
	(Generol 122E-5, sold by Henkel Corp.)	1.00
15		
	<u>Water Phase</u>	
	Deionized Water	55.7512
	Fluilan AWS	3.000
20	Guanidine Carbonate	6.500
	Succinic Acid	0.250
	Tropaeolin "O" (Also known as, Acid Orange 6, sold by Pylam)	0.0015
	Crodafos SG	1.500

25 The pH of the relaxer base is 10.20.

 The guanidine carbonate is present in the cream base in an amount of 0.0621 grams per milliliter. The density of the cream base is 0.956 grams per milliliter.

30 The relaxer base was prepared in a manner similar to that described in Example 1.

 The activator lotion was the same composition as shown in Example 1.

35 The cream base and activator are mixed under ambient conditions in a weight ratio of 12.5 to 1, and the results are similar to those of Example 1.

 The following illustrates that mixing errors of $\pm 10\%$ when estimating half the contents of either the

cream base of the present invention or the activator of the present invention result in minimal changes in the guanidine carbonate concentration in the mixture, and therefore minimal changes in the guanidine hydroxide concentration of the hair relaxer.

A typical two-component hair relaxer system of the present invention prepared in accordance with this example comprises 250 grams of cream base containing 6.5 weight% guanidine carbonate (0.090 moles) and 20 grams of activator lotion.

When the cream base and activator lotion prepared above are mixed in a weight ratio of 12.5 to 1 as set forth above, the corresponding volume ratio is 262 to 14.

When the full contents are mixed, the concentration of guanidine carbonate in the relaxer when the activator and base are mixed equal Cr (gm/ml).

$$Cr = \frac{V_b \times C_b}{V_a + V_b} = \frac{262 \text{ ml} \times 0.0621 \text{ gm/ml}}{14 \text{ ml} + 262 \text{ ml}} = \frac{16.27}{276} = 0.0590 \text{ g/ml}$$

When half contents are measured accurately and mixed, the concentration of guanidine carbonate is the same.

Because the consumer may make an error of $\pm 10\%$ when estimating half the contents of either the cream base or the activator lotion, the following values could be expected.

First, assuming that the consumer employs 10% by volume less base than the true half content of base and 10% by volume more activator than the true half content of activator, a lower Cr is obtained as follows:

$$Cr = \frac{17.9 \text{ ml} \times 0.0621 \text{ gm/ml}}{7.7 \text{ ml} + 111.9 \text{ ml}} = 0.0583 \text{ g/ml}$$

Next, assuming that the consumer employs 10% by volume more base than the true half content of base and 10% by volume less activator than the true half content of activator, a higher Cr is obtained as follows:

$$Cr = \frac{144.1 \text{ ml} \times 0.0621 \text{ gm/ml}}{6.3 \text{ ml} + 144.1 \text{ ml}} = 0.0595 \text{ g/ml}$$

Thus, even with a $\pm 10\%$ error in the volume of cream base and a $\pm 10\%$ error in the volume of activator, the final concentration of guanidine carbonate in the relaxer is less than about $\pm 2\%$ from the desired value.

On the other hand, for a typical relaxer of the prior art where the cream base contains the calcium hydroxide and the liquid activator contains the guanidine carbonate, the following can be shown for a commercially available relaxer system designed to produce a guanidine carbonate concentration in the relaxer of 0.0646 g/ml. Such a relaxer system is

comprised of the following cream base and liquid activator:

Ca(OH)₂ Containing Cream Base

5		<u>Wt%</u>
	<u>Oil Phase</u>	
10	Cosmowax J (Croda) (Cetearyl Alcohol, Ceteareth-20)	10.00
	Aldol 52 (Sherex Chemical Co., Inc., Duplin, Ohio) (Cetyl Alcohol)	2.00
15	Light Mineral Oil (Witco Corp., Melrose Park, IL) (Mineral Oil)	20.00
	<u>Water Phase</u>	
20	Deionized Water	49.79
	Propylene Glycol	5.00
	Fluilan AWS (Croda) (PPG-12 PEG-65 Lanolin Oil)	3.00
25	Calcium Hydroxide	5.21
	Duponol XL (DEA-Lauryl Sulfate, Sodium Lauramino-propionate, DEA- Lauraminopropionate)	5.00

30 The Ca(OH)₂ containing cream base has a density of 1.013 grams per milliliter, and is used in an amount of 218 grams corresponding to a volume of 215 milliliters.

Guanidine Carbonate Containing Liquid Activator

35		<u>Wt%</u>
	Deionized Water	75.49
	Keltrol (Kelco, San Diego, CA) (Xanthan Gum)	0.50
40	Guanidine Carbonate	24.00
	FD&C Yellow #5	0.007
	FD&C Red #4	0.005

The guanidine carbonate is present in the liquid activator in an amount of 0.2611 grams per milliliter, and the activator has a density of 1.088 g/ml. The liquid activator is used in an amount of 77 grams, corresponding to a volume of 71 milliliters.

Thus, when the full contents are mixed the concentration of guanidine carbonate in the relaxer (Cr) is as follows:

$$Cr = \frac{V_a \times C_a}{V_a + V_b} = \frac{71 \text{ ml} \times 0.2611 \text{ gm/ml}}{71 \text{ ml} + 215 \text{ ml}} = 0.0646 \text{ g/ml}$$

When half contents are measured accurately and mixed, the concentration of guanidine carbonate is the same:

$$Cr = \frac{35.5 \text{ ml} \times 0.2611 \text{ gm/ml}}{35.5 \text{ ml} + 107.5 \text{ ml}} = 0.0646 \text{ g/ml}$$

Because the consumer may make an error of $\pm 10\%$ when estimating half of the contents of either the cream base or the liquid activator, the following values could be expected.

First, assuming that the consumer employs 10% by volume less base than the true half contents of base and 10% by volume more activator than the true half contents of activator, a higher Cr is obtained as follows:

$$Cr = \frac{39.05 \text{ ml} \times 0.2611 \text{ gm/ml}}{39.05 \text{ ml} + 96.25 \text{ ml}} = 0.0749 \text{ g/ml}$$

Next, assuming that the consumer employs 10% by volume more base and 10% by volume less activator than the true half contents, a lower Cr is obtained as follows:

5 Cr = $\frac{31.95 \text{ ml} \times 0.2611 \text{ gm/ml}}{31.95 \text{ ml} + 118.25 \text{ ml}}$ = 0.0554 g/ml

Thus, with a $\pm 10\%$ error in the volume of cream base and a $\pm 10\%$ error in the volume of activator, the final concentration of guanidine carbonate in the relaxer is approximately $\pm 15\%$ from the desired value. Such a variation in the strength of the relaxer is significant and can led to highly over-processed hair with an increase in hair damage and irritation or highly underprocessed hair.

A group of 25 women who were currently users of the above commercially available product reported significantly less tingling, stinging and burning on the skin and scalp with a relaxer composition prepared in accordance with this example than with the commercially available product they had been employing.

EXAMPLE 3

25 A two-component hair relaxer system according to the present invention was formulated, with one component being in the form of a cream base and the

other component being in the form of a powder. The components had the following composition.

Formulation of Relaxer Base Containing Guanidine Carbonate.

5

Weight %

Oil Phase

10	Cetyl alcohol	4.80
	Stearyl alcohol	5.20
	Steareth-20	1.00
	Mineral oil	10.00
	Petrolatum	10.00
15	Steareth-10	1.00

Water Phase

	Deionized Water	59.01
20	Fluilan AWS	3.00
	Disodium EDTA	0.20
	Guanidine Carbonate	5.59
	Succinic Acid	0.2

25 The pH of the relaxer base is 10.28.

The relaxer base was prepared in a manner similar to that described in Example 1.

The activator powder has the following composition and was prepared by wet/dry blending followed by sieving with a 200 mesh sieve.

30

Wt. %

	Calcium Hydroxide powder	88.0
	Caprylyl Pyrrolidone	
	(Surfadone LP100	12.0
35	sold by ISP Inc.)	
		<u>100.0</u>

The cream base and activator powder are mixed under ambient conditions in a weight ratio of 20 to

1, and the results are similar to those of Example 1.

EXAMPLE 4

A two-component hair relaxer system according to the present invention was formulated, with one component being in the form of a cream base containing a non-polymeric conditioner, and the other component being in the form of a powder.

Formulation of Relaxer Base Containing Guanidine Carbonate.

<u>Ingredient</u>	<u>Weight %</u>
<u>Oil Phase</u>	
Cetyl alcohol	3.52
Stearyl alcohol	3.81
Steareth-20	0.74
Mineral oil	9.74
Petrolatum	9.74
Steareth-10	1.00
<u>Water Phase</u>	
Deionized Water	58.92
Fluilan AWS	2.46
Arquad 2HT-75	1.50
(Quaternium-18 and Isopropyl alcohol)	
Guanidine Carbonate	6.99
Succinic Acid	0.2
Crodafos SG	1.64

The pH of the relaxer base is 10.28.

The relaxer base was prepared in a manner similar to that described in Example 1.

The activator was commercial powdered calcium hydroxide.

5 The cream base and activator are mixed under ambient conditions in a weight ratio of 24 to 1, and the results are similar to those of Example 1.

The relaxer is tested for efficacy of relaxation and reversion according to the following procedure.

10 Swatches of hair are prepared containing 20 to 40 hairs bound at the proximal end with waterproof tape.

For each test, 3 swatches are attached to the surface of a glass plate.

15 The relaxer is applied to the hair, using long strokes to fully extend the hair. Each swatch is stroked with a soft camel's hair brush every 5 to 10 minutes until the relaxer has been on the hair for the appropriate amount of time for the relaxer which
20 is being tested.

The relaxer is then rinsed from the hair and glass plate with tap water.

A 50:50 blend of neutralizing shampoo and water then is carefully applied to the hair. The neutral-
25 izing shampoo mixture is kept in constant contact with the hair for at least 3 minutes. Each swatch

is rinsed with water to remove the neutralizing shampoo.

Each swatch is attached loosely to a glass rod with a cotton thread and dried with a hair dryer set on cool for 1 hour.

The swatches are then removed from the glass rods. The bound end of the relaxed swatches is attached to alligator clips positioned at a zero line on a finely divided graph paper. The graphs are placed in a vertical position and equilibrated to room temperature and 65% relative humidity (RH) for 1 hour.

A pressure sensitive label is attached to the hair so that the upper edge of the label marks a point that is 10 to 15 cm from the bottom of the tape that binds the swatch. This point is recorded as the initial length (L_r).

With the label holding hairs together, the hair is gently pulled until it is fully extended but not stretched. The point marked by the upper edge of the label used to hold the hair is recorded as the extended length (L_s).

The hair is then cut along the upper edge of the label.

Each graph is placed in a vertical position inside a constant-humidity chamber maintained at 90% RH for 24 hours.

After 24 hours, each graph is removed from the humidity chamber and the swatches are allowed to equilibrate to room temperature and 65% RH for 1 hour. The length of each swatch (L_v) is recorded without touching the hair.

The % relaxation and % reversion are calculated as follows:

$$1) \quad \% \text{ Relaxation} = 100 \times \left(1 - \frac{L_s - L_r}{L_s} \right)$$

where L_s = extended length
 L_r = original length

$$2) \quad \% \text{ Reversion} = \left[\% \text{ relaxation} - \left(100 \times \left(1 - \frac{L_s - L_c}{L_s} \right) \right) \right]$$

where L_s = initial extended length
 L_c = length after reversion

The relaxer was subjected to two relaxation/-reversion tests, and the data for each test is shown below.

	<u>Relaxation %</u>	<u>Reversion %</u>
Test No. 1	97.8 ± 0.0	1.1 ± 1.1
Test No. 2	98.5 ± 0.5	0.0 ± 0.0

EXAMPLE 5

A two-component hair relaxer system according to the present invention was formulated with one component being in the form of a cream base containing a polymeric conditioner, and the other component being in the form of commercial calcium hydroxide powder.

The components had the following composition.

Formulation for Relaxer Base Containing Guanidine Carbonate without Acidulant/buffer and without color indicator

Ingredient	Wt%
<u>Oil Phase</u>	
Cetyl alcohol	3.26
Stearyl alcohol	3.53
Steareth-10	0.68
Steareth-20	0.68
Mineral oil	8.00
Petrolatum	8.00
<u>Water Phase</u>	
Water	55.78
Propylene glycol	3.77
Fluilan AWS	2.28
Guanidine carbonate	5.59
Duponol XL	1.51
Polyquaternium-2 (Mirapol A-15, sold by Miranol Inc.)	3.02

In preparing the above relaxer base composition, the oil phase is prepared as in Example 1. The water, propylene glycol and Fluilan AWS are heated quickly to 70°C, and the heated oil phase is

then added to these ingredients slowly with moderate homogenization. The mixture is left to cool, and when the mixture reaches 60°C, homogenizing is continued while Mirapol A-15 is added. When the mixture reaches 45°C, guanidine carbonate is added.

The activator comprises commercial powdered calcium hydroxide.

To form the hair relaxer, the cream base and the activator are admixed under ambient conditions in a weight ratio of 96:4 of cream base to activator.

Example 7

The procedure of Example 6 was repeated, except that various acids as shown in the Table below were added to the relaxer base formulation of Example 6.

A mole ratio of 0.1:1 acid to guanidine carbonate was maintained for each relaxer base.

The pH of the relaxer base was determined based on a 10-minute immersion of a gel-filled combination electrode in the relaxer base formulation.

Each relaxer base was tested for efficacy of relaxation and reversion according to the procedure set forth in Example 5 above.

EVALUATION OF RELAXER-BASE FORMULATIONS CONTAINING GUANIDINE CARBONATE AND VARIOUS ACIDS

Sample No.	Proton Source	pH	Relaxation%	Reversion%
1	---	11.0	97.8 \pm 0.0	5.8 \pm 5.4
2	Lactic acid	10.7	97.0 \pm 0.5	5.8 \pm 3.1
3	Tartaric acid	10.3	96.5 \pm .02	11.4 \pm 5.8
4	Ascorbic acid	10.3	95.1 \pm 0.3	12.0 \pm 6.4
5	Maleic acid	10.3	97.2 \pm 0.3	1.3 \pm 1.1
6	Glutamic acid	10.4	97.7 \pm 0.2	4.9 \pm 1.6
7	Succinic acid	10.4	97.4 \pm 0.6	3.7 \pm 2.4

The headspace above Sample 1 had a slight ammonia odor. This odor became more intense when the formulation was stirred. In contrast, no ammonia odor was noted in Sample Nos. 2 to 7.

Samples 3 and 4 had an acceptable relaxation efficacy, but reversion for these samples was more than 10%. Although these samples provide acceptable relaxation, they are not preferred because of their higher reversion. Samples 5 to 7 provided good relaxation and low reversion.

EXAMPLE 8

A liquid activator containing guanidine carbonate is prepared for use with a $\text{Ca}(\text{OH})_2$ containing

cream base. The liquid activator has the following composition.

	Guanidine carbonate	25 gm
	Succinic Acid	1.64 gm
5	Water	73.36 gm

EXAMPLE 9

A liquid activator containing guanidine carbonate is prepared for use with a $\text{Ca}(\text{OH})_2$ containing cream base. The liquid activator has the following composition.

	Guanidine carbonate	25 gm
	Glutamic acid	2.04 gm
15	Water	72.96 gm

It will be understood that the above description of the present invention is susceptible to various modifications, changes and adaption, and the same are intended to be comprehended within the meaning and range of equivalents of the appended claims.

WHAT IS CLAIMED IS:

1. A two-component hair relaxer system for producing an alkaline hydroxide relaxer for relaxing hair comprising:

(a) a first component comprising a cream base containing a water-soluble salt of a relatively strong base with an anion capable of being precipitated by an alkaline earth metal ion under highly alkaline conditions, and

5 (b) a second, separate component, which is substantially free of water, and containing an alkaline material having an alkaline earth metal ion which forms a precipitate with the anion when the first component and second component are mixed.

2. The relaxer system according to claim 1, wherein the relatively strong base is selected from the group consisting of guanidine, N-methyl guanidine, dimethylaminoguanidine, acetamide, ~~dimethylamino-~~amidine, aminoamidine and acetamide.

3. The relaxer system according to claim 1, wherein the anion of the water-soluble salt is selected from the group consisting of carbonate, sulfate, sulfite, phosphate, fluoride, oxalate, tartrate, laurate and alginate.

4. The relaxer system of claim 1, wherein the water-soluble salt is guanidine carbonate.
5. The relaxer system of claim 1, wherein the water-soluble salt is present in the cream base in an amount of 1 to 20% by weight of the cream base.
6. The relaxer system according to claim 1, wherein the water-soluble salt is present in the cream base in an amount of from about 1 to about 10% by weight of the cream base.
7. The relaxer system according to claim 1, wherein the water-soluble salt is present in the cream base in an amount of from about 6 to about 8% by weight of the cream base.
8. The relaxer system according to claim 1, wherein the cream base contains a conditioning agent.
9. The relaxer system according to claim 1, wherein the conditioning agent is present in an amount of about 0.1 to about 4% by weight of the cream base.

10. The relaxer system according to claim 1, wherein the conditioning agent is a non-polymeric quaternary nitrogen containing component.

11. The relaxer system according to claim 1, wherein the alkaline material is an alkaline earth metal hydroxide, an alkaline earth metal oxide, or a mixture thereof.

12. The relaxer system according to claim 1, wherein the alkaline material is calcium hydroxide, calcium oxide or a mixture thereof.

13. The relaxer system according to claim 1, wherein the second component contains less than about 20 weight percent water.

14. The relaxer system according to claim 1, wherein the second component contains less than 10% by weight water.

15. The relaxer system according to claim 1, wherein the second component is anhydrous.

16. The relaxer system according to claim 1, wherein the second component is a pourable suspension

which contains 20 to 60 weight percent of the alkaline material.

17. The relaxer system according to claim 1, wherein the second component contains a water-miscible, hydrophilic liquid carrier.

18. The relaxer system according to claim 1, wherein the liquid carrier is a polyhydroxy compound or an ether.

19. The relaxer system according to claim 1, wherein the liquid carrier is selected from propylene glycol, glycerine, butylene glycol and hexylene glycol.

20. The relaxer system according to claim 1, wherein the second component is a powder.

21. The relaxer system according to claim 1, wherein the alkaline material is present in the powder in an amount of 20 to 100% by weight of the powder.

22. The relaxer system according to claim 1, wherein the alkaline material is present in the second component in an amount sufficient to provide a molar

excess of from 5 to 200% with respect to the water-soluble salt of the cream base.

23. The relaxer system according to claim 1, wherein the volume ratio of the second component to the first component is from one part by volume second component to about 8 to 50 parts by volume cream base.

24. The relaxer system according to claim 23, wherein the volume ratio of the second component to the first component is from one part by volume second component to about 10 to 25 parts by volume cream base.

25. The relaxer system according to claim 1, wherein the density of the second component is from about 1.4 gm/ml to about 2.34 gm/ml.

26. The relaxer system according to claim 1, wherein the weight ratio of second component to first component is from one part by weight second component to about 6 to 30 parts by weight cream base.

27. The relaxer system according to claim 1, wherein the weight ratio of second component to first

component is from one part by weight second component to about 10 to 15 parts by weight cream base.

28. The relaxer system according to claim 1, wherein the water-soluble salt is a salt of relatively strong organic nitrogen containing base with a carbonate anion and which is susceptible to alkaline hydrolysis and formation of ammonia, and wherein the first component contains a carbonate/bicarbonate buffer to retard decomposition of the water-soluble salt and formation of ammonia.

29. The relaxer system according to claim 28, wherein the buffer provides the cream base with a pH of from about 9.5 to about 10.5.

30. The relaxer system according to claim 29, wherein an acid which does not interfere with the relaxing is present in the cream base to form the buffer and provide the pH of from about 9.5 to about 10.5.

31. The relaxer system according to claim 30, wherein the acid is one which does not cause a degree of reversion of greater than about 15%.

32. The relaxer system according to claim 30, wherein the acid is an organic acid.

33. The relaxer system according to claim 30, wherein the acid is at least one acid selected from the group consisting of an aliphatic acid, an alicyclic acid, an aromatic acid and a heterocyclic acid.

34. The relaxer system according to claim 30, wherein the acid is at least one acid selected from the group consisting of a monocarboxylic acid and a dicarboxylic acid.

35. The relaxer system according to claim 30, wherein the acid is at least one acid selected from an aliphatic monocarboxylic acid and an aliphatic dicarboxylic acid.

36. The relaxer system according to claim 30, wherein the acid is at least one acid selected from the group consisting of succinic acid, maleic acid, oxalic acid, glutamic acid and lactic acid.

37. The relaxer system according to claim 30, wherein the acid is at least one acid selected from

the group consisting of succinic, maleic acid, and glutamic acid.

38. The relaxer system according to claim 30, wherein the acid is succinic acid.

39. The relaxer system according to claim 30, wherein the acid is glutamic acid.

40. The relaxer system according to claim 30, wherein the acid is an inorganic acid.

41. The relaxer system according to claim 30, wherein the acid is sulfuric acid or phosphoric acid.

42. The relaxer system according to claim 30, wherein the acid contains at least one substituent selected from an amino group and a hydroxy group.

43. The relaxer system according to claim 30, wherein the acid is present in an amount of from about 2 to 20 mol% of the number of mols of water-soluble salt.

44. The relaxer system according to claim 30, wherein the acid is present in an amount of from 5 to 10 mol% of the number of mols of water-soluble salt.

45. The relaxer system according to claim 29, wherein the first component contains a pH indicator.

46. The relaxer system according to claim 45, wherein the pH indicator is present in an amount of from 0.0005 to 0.5% by weight of the first component.

47. The relaxer system according to claim 45, wherein the pH indicator is selected from the group consisting of Tropaeolin "O", Thiazole Yellow "G", Eosin I, Pourriers Blue, Mordant Blue #1, Mordant Red #3 and External D&C Violet #2.

48. A water-containing composition which contains a water-soluble salt of a relatively strong organic nitrogen containing base with a carbonate anion and which is susceptible to alkaline hydrolysis and formation of ammonia, wherein the water-containing composition contains a carbonate/bicarbonate buffer

to retard decomposition of the water-soluble salt and formation of ammonia.

49. The composition according to claim 48, wherein the relatively strong base is selected from the group consisting of guanidine, N-methyl guanidine, dimethylaminoguanidine, acetamide, dimethylaminoamidine, aminoamidine and acetamine.

50. The composition according to claim 48, wherein the water-soluble salt is guanidine carbonate.

51. The composition according to claim 48, wherein the composition is in the form of a cream base and the water-soluble salt is present in the cream base in an amount of 1 to 20 % by weight of the cream base.

52. The composition according to claim 51, wherein the water-soluble salt is present in the cream base in an amount of from about 1 to about 10% by weight of the cream base.

53. The composition according to claim 51, wherein the water-soluble salt is present in the cream base

in an amount of from about 6 to about 8% by weight of the cream base.

54. The composition according to claim 48, wherein the water-containing composition is a water solution containing about 20 to 30% by weight of the water-soluble salt.

55. The composition according to claim 54, wherein the water-containing composition is a liquid activator for use in combination with a hair relaxer cream base which contains an alkaline material having an alkaline earth metal ion which forms a precipitate with the carbonate anion, the activator comprising a water-solution of said water-soluble salt, said activator, when subsequently mixed with the cream base reacting with the alkaline material to produce a hair relaxer composition.

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56. The composition according to claim 48, wherein the buffer provides a pH of from about 9.5 to about 10.5.

57. The composition according to claim 48, wherein an acid which does not interfere with the relaxing

process is present to form the buffer and provide a pH of from about 9.5 to about 10.5.

58. The composition according to claim 57, wherein the acid is one which does not cause a degree of reversion of greater than about 15%.

59. The composition according to claim 57, wherein the acid is an organic acid.

60. The composition according to claim 57, wherein the acid is at least one acid selected from the group consisting of an aliphatic acid, an alicyclic acid, an aromatic acid and a heterocyclic acid.

61. The composition according to claim 57, wherein the acid is at least one acid selected from the group consisting of an aliphatic monocarboxylic acid and an aliphatic dicarboxylic acid.

62. The composition according to claim 57, wherein the acid is at least one acid selected from the group consisting of succinic acid, maleic acid, oxalic acid, glutamic acid, and lactic acid.

63. The composition according to claim 57, wherein the acid is at least one acid selected from succinic acid, maleic acid and glutamic acid.

64. The composition according to claim 57, wherein the acid is succinic acid.

65. The composition according to claim 57, wherein the acid is glutamic acid.

66. The composition according to claim 57, wherein the acid is an inorganic acid.

67. The composition according to claim 57, wherein the acid is sulfuric acid or phosphoric acid.

68. The composition according to claim 57, wherein the acid is present in an amount of from about 2 mol% to about 20 mol% based on the number of moles of water-soluble salt present in the composition.

69. The composition according to claim 57, wherein the acid is present in an amount of from about 5 to 10 mol% based on the number of moles of water-soluble salt present in the composition.

70. The composition according to claim 57, wherein the composition contains a pH indicator.

71. The composition according to claim 70, wherein the pH indicator is present in an amount of from 0.0005 to 0.5% by weight of the composition.

72. The composition according to claim 70, wherein the pH indicator is selected from the group consisting of Tropaeolin "O", Thiazole Yellow "G", Eosin I, Poirriers Blue, Mordant Blue #1, Mordant Red #3 and External D&C Violet #2.

73. An activator lotion for use in combination with a hair relaxer cream base, which cream base contains a water-soluble salt of a relatively strong base with an anion capable of being precipitated by an alkaline earth metal ion under highly alkaline conditions, the lotion being substantially free of water and comprising (a) a suspension of an alkaline material having an alkaline earth metal ion which forms a precipitate with the anion when the cream base and lotion are mixed and (b) a water-miscible, hydrophilic liquid carrier.

74. The activator lotion according to claim 73, wherein the alkaline material is present in an amount of from 20 to 60 weight percent of the lotion.

75. The activator lotion according to claim 73, wherein the alkaline material is an alkaline earth metal hydroxide, an alkaline earth metal oxide, or a mixture thereof.

76. The activator lotion according to claim 73, wherein the alkaline material is calcium hydroxide, calcium oxide or a mixture thereof.

77. The activator lotion according to claim 73, wherein the lotion contains less than 20% by weight water.

78. The activator lotion according to claim 73, wherein the lotion contains less than 10% by weight water.

79. The activator lotion according to claim 73, wherein the lotion is anhydrous.

80. The activator lotion according to claim 73, wherein the lotion contains a thickening agent.

81. The activator lotion according to claim 73, wherein the liquid carrier is a polyhydroxy compound or an ether.

82. The activator lotion according to claim 73, wherein the liquid carrier is selected from propylene glycol, glycerine, butylene glycol and hexylene glycol.

83. A process for producing a hair relaxer composition for relaxing hair comprising mixing the first component and second component of claim 1.

84. A hair relaxer composition comprising the mixture resulting from admixing the first component and second component of claim 28.

85. A method for relaxing hair comprising the steps of:

(a) preparing a hair relaxer by admixing the first component and second component of claim 1,

(b) applying to the hair an effective amount of the hair relaxer prepared in step (a),

(c) smoothing the hair relaxer on the hair and leaving the hair relaxer in the hair for a time sufficient to achieve adequate hair relaxing, and

(d) removing the hair relaxer from the hair.

86. A method for relaxing hair according to claim 85, wherein the water soluble salt is guanidine carbonate, the alkaline material is calcium hydroxide, and the weight ratio of the second component to the first component is from one part by weight second component to about 6 to 30 parts by weight of the first component.

87. A method for relaxing hair according to claim 85, wherein the first component contains a carbonate/bicarbonate buffer to retard decomposition of the guanidine carbonate and formation of ammonia.

88. A method for relaxing hair according to claim 85, wherein the buffer provides the cream base with a pH of from about 9.5 to about 10.5.

89. The method for relaxing hair according to claim 85, wherein an acid which does not interfere with the relaxing process is present in the cream base to

form the buffer and provide the pH of from about 9.5 to about 10.5.

90. The method for relaxing hair according to claim 89, wherein the acid is at least one acid selected from the group consisting of an aliphatic monocarboxylic acid and an aliphatic dicarboxylic acid.

91. The method for relaxing hair according to claim 89, wherein the acid is at least one acid selected from the group consisting of succinic acid, maleic acid and glutamic acid.

92. The method for relaxing hair according to claim 89, wherein the first component contains a pH indicator.

93. A two-component hair relaxing system for producing an alkaline hydroxide relaxer for relaxing hair comprising:

(a) a first component comprising a liquid activator comprising a water-solution of a water-soluble salt of a relatively strong organic nitrogen containing base with a carbonate anion and which is susceptible to alkaline hydrolysis and formation of ammonia, wherein the water-solution contains a car-

bonate/bicarbonate buffer to retard decomposition of the water-soluble salt and formation of ammonia, and

- 5 (b) a cream base which contains an alkaline material having an alkaline earth metal ion which forms a precipitate with the carbonate anion, the first component when subsequently mixed with the second component reacting with the alkaline material to produce a hair relaxer composition.

94. A two-component hair relaxing system according to claim 93, wherein the water-soluble salt is guanidine carbonate and the alkaline material is calcium hydroxide.

95. A two-component hair relaxing system according to claim 93, wherein the water-soluble salt is present in the first component in an amount of about 20 to about 30% by weight of the first component, and the alkaline material is present in the second component in an amount of about 4 to about 7% by weight of the second component.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/07813

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : A61K 7/09, 7/08

US CL : 424/70, 71, 72; 132/7

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/70, 71, 72; 132/7

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Cosmetic and toiletry formulations Vols 1 and 2 by Ernest W. Flick

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 4,304,244 (DE LA GUARDIA) 08 December 1981, Abstract, columns 2-4, columns 7-10, Examples.	1-95

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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